



Article The Preparation of Ground Blast Furnace Slag-Steel Slag Pavement Concrete Using Different Activators and Its Performance Investigation

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Abstract: Steel slag and ground blast furnace slag show good wear resistance, which is suitable for improving the abrasion performance of pavement concrete. This work presents an investigation of the activation of Na_2SO_4 , Na_2CO_3 and Na_2SiO_3 on the GBFS-SS composite pavement concrete. The results showed that both Na₂SO₄ and Na₂SiO₃ can promote the strength development of the GBFS-SS composite cementitious system. Na₂CO₃ shows limited improvement in the strength of GBFS-SS composite paste. The GBFS-SS composite paste activated with Na2SiO3 and Na2SO4 combination shows hydration products of ettringite, portlandite and amorphous C-A-S-H gel. SO_4^{2-} can accelerate the depolymerization of the aluminosilicate network in GBFS and SS vitreous structure, while SiO_3^{2-} can only facilitate the pozzolanic reaction of GBFS and SS, but also participate in the hydration to form more C-A-S-H gel. Na₂SO₄ as the activator can reduce the dry shrinkage of the pavement concrete, while Na₂SiO₃ as the activator can further improve the compressive strength and abrasion resistance of the pavement concrete. The combined activation of Na₂SiO₃ and Na₂SO₄ shows a better effect on improving the performance of pavement concrete than the single Na_2SiO_3 or Na2SO4 activator. At the optimal content of 3% of Na2SiO3 and 1% of Na2SO4, the pavement concrete obtains the 60 d compressive strength of 73.5 MPa, the 60 d drying shrinkage of 270×10^{-6} , the 60 d interconnected porosity of 6.85%, and the 28 d abrasion resistance of $28.32 \text{ h}/(\text{kg/m}^2)$.

Keywords: steel slag; combined activation; pavement concrete; compressive strength; abrasion resistance

1. Introduction

As the main raw materials used in the construction industry, the production process of cement clinker requires a "two grinding and one burning" process, and the cost of resources and energy is huge (for every 1 t of cement clinker, 1.2 t of limestone, 0.18 t of clay and 0.15 t of standard coal are consumed, and 0.85 t of carbon dioxide gas will be emitted into the air [1]). It goes against the low-carbon green building concept advocated by the world. To this end, scholars have studied the use of industrial solid waste to replace part of the cement used to prepare concrete, such as fly ash [2,3] and slag [4,5], etc., and some scholars have also started to explore the use of cement clinker free alkali-activated materials as concrete materials [6–9]. After long-term exploration and practice, fly ash, slag and so on have been realized as high-quality resource utilization in the construction industry. They have been transformed from bulk industrial wastes into supplementary cementing materials. Compared with the wide application of fly ash and slag, steel slag and other bulk industrial solid wastes are very limited in the building materials industry [10].

Steel slag is a by-product of the metallurgy industry, whose emissions account for about 15–20% of the iron and steel output [11–13]. At present, the comprehensive utilization rate of steel slag in China is low, which is only about 20% [14], and the output of steel slag



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Copyright: © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). has been increasing rapidly with the development of the iron and steel industry. Up to now, the waste steel slag accumulated in China has exceeded 400 million tons, with a 100 million tons increment per year [15]. The discharge and accumulation of steel slag not only occupy a large amount of land, but also pollute the ecological environment due to the alkaline leachates. Building materials are the most effective way to absorb industrial solid waste on a large scale at the present stage [16-18]. The basic reactive minerals in steel slag are tricalcium silicate (C_3S), dicalcium silicate (C_2S) and dicalcium ferrite (C_2F) [19], and these mineral components make steel slag have certain cementitious capabilities [20], but the C_3S and C_2S in steel slag are presented in coarse crystals, with dense structure and low reactivity [21]. Steel slag can hydrate in water at ambient temperature curing conditions, but its hydration rate is very slow and the induction period is very long [22]. Although the hydration products of steel slag are the same as the main hydration products of Portland cement, which are C-S-H gel and Ca(OH)₂ [23], the cohesive force of the hardened steel slag paste is very low. The aluminate minerals in steel slag hydrate later than silicate minerals, and the inert components like the RO phase do not participate in the hydration reaction and only play as the fillers embedded in hydration products in the early hydration stage [24,25].

Steel slag and ground blast furnace slag (GBFS) have similar composition and vitreous structure, high content of Fe and Mn and high wear resistance, which can be applied to the pavement concrete preparation to improve its abrasion performance [26–28]. Cao et al. [29] studied the mechanical properties and microstructure of slaked lime carbonated steel slag mixture and found that slaked lime can enhance the hydration of steel slag powder. The investigation from Zhao et al. [22] revealed that the hydration product of steel slag is C-S-H gels and $Ca(OH)_2$. Their study also proves that a 5% gypsum addition can improve the hydration degree of steel slag. Furthermore, its hydration can also be activated by elevated temperature [30]. Phoo-Ngernkham et al. [31] studied the effect of sodium hydroxide and water glass on the fly ash-GBFS geopolymer. They found that the compressive strength and microstructure of the geopolymer paste improved with the increased content of GBFS. Nguyen et al. [32] found that the 1 d and 28 d compressive strength of 50%slag-50%cement blend activated with 1% Na2SO4 was 5 MPa and 46 MPa, respectively. The slag cement blend activated with 5% and 10% Na₂SO₄ has 28 d compressive strength of 36~56 MPa, respectively. In addition, the volume shrinkage of the Portland slag cement sample decreased with the increase of Na₂SO₄ content. If reasonable measures can take to activate the reactivity of steel slag, the $Ca(OH)_2$ generated by its hydration can be used to promote the pozzolanic reaction of GBFS in the cementitious system. The combined activation of these solid wastes and their high-volume application in pavement concrete can be realized. This can significantly reduce the cost of pavement concrete and improve its abrasion performance. However, the effect of activator type on the performance of steel slag-GBFS composite was not fully understood.

Therefore, this work intends to use GBFS and steel slag as cementitious materials to produce pavement concrete. Different activators were selected to promote the pozzolanic reaction of steel slag and GBFS. The influence of the activator on the mechanical properties of the blended pastes was studied to optimize the incorporation content of the activators and its activation mechanisms were revealed [33]. Subsequently, the mechanical properties, abrasion performance, volume stability and durability of the GBFS-SS composite concrete were studied.

2. Raw Materials and Methods

2.1. Raw Materials

The main cementitious materials used in this paper are Portland cement, granulated blast furnace slag (GBFS) and converter steel slag (SS). Cement is made from Hubei Yadong Cement Co., Ltd. (Wuhan, China) of strength grade of P·II52.5, density of 3.06 g/cm³ and specific surface area of 360 m²/kg. Steel slag is produced in Taizhen Mineral Products Processing Plant (Shijiazhuang, China), density of 3.27 g/cm³ and specific surface area of 480 m²/kg. The XRD patterns of SS, cement and GBFS are shown in Figure 1. The

main phases in steel slag are C_2S , C_3S , C_2F and RO phase (RO phase is a solid solution of FeO, MgO and MnO). GBFS is produced in Lingshou County, Shijiazhuang City, Hebei Province, which is graded as S95 according to Chinese GB/T 18046-2017 Standard [34]. The density and specific surface area of GBFS are 2.92 g/cm³ and 450 m²/kg, respectively. The chemical compositions for cement, GBFS and steel slag powder from X-ray fluorescence spectrometer (XRF) tests are shown in Table 1.



Figure 1. XRD pattern of SS, cement and GBFS.

| Table 1. Chemical compositions of cement, GBFS and SS (wt. | .% | 5) |
|--|----|----|
|--|----|----|

| Raw Material | SiO ₂ | Al ₂ O ₃ | CaO | Fe ₂ O ₃ | MgO | MnO | P_2O_5 | SO ₃ | Other | Loss of Ignition |
|-----------------|------------------|--------------------------------|-------|--------------------------------|------|------|----------|-----------------|-------|---------------------|
| SS | 14.74 | 6.51 | 36.13 | 15.18 | 9.21 | 2.73 | 1.16 | 1.05 | 8.42 | 4.87 |
| GBFS | 31.03 | 14.92 | 40.85 | 0.27 | 8.03 | 0.47 | 0.01 | 2.48 | 1.60 | 0.34 |
| Cement | 20.24 | 4.11 | 62.92 | 3.06 | 2.54 | 0.07 | 0.14 | 2.31 | 1.43 | 3.18 |

The particle size distribution of the cementitious materials was determined by Mastersizer2000 laser particle size analyzer (Malvern Panalytical, UK), and the results are shown in Figure 2. As can be seen from the figure, the most probable particle sizes are 17 μ m for GBFS, 21 μ m for cement and 53 μ m for SS. This means GBFS and cement have similar particle size distribution, SS shows the coarse particle size distribution, which is mainly concentrated in 18 μ m~200 μ m.

The coarse aggregate used in this work is 5–10 mm and 10–20 mm basalt crushed stones, provided by Yichang Yuyuan Construction Co., Ltd. (Yichang, China), with a density of 3.08 g/cm^3 , crushing value of 8.3%, Los Angeles wear value of 12.5% (Los Angeles wear value defined by the ASTM C131 Standard [35]), water absorption of 0.52% and needle flake content of 6.2%. The grain grading of coarse aggregates is shown in Tables 2 and 3. In pavement concrete preparation, the weight ratio of 5–10 mm and 10–20 mm basalt crushed stones is 4:6.



Figure 2. Particle size distribution of cementitious materials. (**a**) Particle size distribution; (**b**) cumulative particle size distribution.

| Table 2. Grain gradi | ng of 5–10 mm l | basalt crushed | stones. |
|----------------------|-----------------|----------------|---------|
|----------------------|-----------------|----------------|---------|

| Aperture Size/mm | 0~2.36 | 2.36~4.75 | 4.75~9.50 | 9.50~13.2 |
|------------------|--------|-----------|-----------|-----------|
| Grading/% | 0.4 | 6.1 | 92.0 | 1.5 |

Table 3. Grain grading of 10-20 mm basalt crushed stones.

| Aperture Size/mm | 0~2.36 | 2.36~4.75 | 4.75~9.50 | 9.50~13.2 | 13.2~16.0 | 16.0~19.0 |
|------------------|--------|-----------|-----------|-----------|-----------|-----------|
| Grading/% | 0.2 | 0.2 | 7.9 | 83.9 | 4.6 | 3.2 |

High titanium heavy slag (HTHS) sand is used as fine aggregate. It has a packing density of 3100 kg/m^3 , an apparent density of 1925 kg/m^3 and a fineness modulus of 3.24. The grain grading of fine aggregate is shown in Table 4. The water content of HTHS is 8.4%. Due to the highly interconnected pores in high titanium heavy slag sand, it can play as an internal curing material in concrete.

Table 4. Grain grading of high titanium heavy slag sand.

| Aperture Size/mm | <0.15 | 0.15~0.3 | 0.3~0.6 | 0.6~1.18 | 1.18~2.36 | 2.36~4.75 | ≥4.75 |
|------------------|-------|----------|---------|----------|-----------|-----------|-------|
| Grading/% | 1.7 | 4.5 | 23.3 | 25.7 | 28.2 | 16.6 | 0 |

Polycarboxylic acid superplasticizer produced by Jiangsu Subote New Materials Co., Ltd. (Nanjing, China), was used in this experiment, which has a water reduction rate greater than 20%. Na₂SO₄, Na₂CO₃ and Na₂SiO₃ activators are analytical grade reagents produced by Tianjin Beichen Founder Reagent Factory. The retarder is commercial borax (Na₂B₄O₇·10H₂O).

2.2. Mixing Proportion

The optimal mixing proportion of blended paste was determined to be steel slag:GBFS:cement = 36:54:10, through previous experiments. The effect of single and combination activation of three types of activators (Na₂SO₄, Na₂CO₃ and Na₂SiO₃) on the performance of the paste was studied. In the control group, the water to binder (w/b) ratio was 0.25 and the content of Superplasticizer was 2%. In the activator-containing groups, the w/b ratio was set at 0.28 and the content of the water-reducing agent was 3.2%. At the same time, 0.2% borax was added as a retarder when the activator was added [36]. The

detailed mixing proportion was given in Table 5, where the control group was named C. The activated group was named as the activator type and its percentage, such as the S1 group indicating 1% of Na₂SO₄ addition. Note that the content of activators was calculated according to their Na₂O content by the weight of total cementitious materials.

| No. | SS | GBFS | Cement | Na ₂ SO ₄ | Na ₂ CO ₃ | Na_2SiO_3 | Borax | w/b Ratio | Superplasticizer |
|-----|----|------|--------|---------------------------------|---------------------------------|-------------|-------|--------------|------------------|
| С | 36 | 54 | 10 | - | - | - | - | 0.25 | 3.2 |
| S1 | 36 | 54 | 10 | 1 | - | - | 0.2 | 0.28 | 3.2 |
| S2 | 36 | 54 | 10 | 2 | - | - | 0.2 | 0.28 | 3.2 |
| S3 | 36 | 54 | 10 | 3 | - | - | 0.2 | 0.28 | 3.2 |
| S4 | 36 | 54 | 10 | 4 | - | - | 0.2 | 0.28 | 3.2 |
| S5 | 36 | 54 | 10 | 5 | - | - | 0.2 | 0.28 | 3.2 |
| C1 | 36 | 54 | 10 | - | 1 | - | 0.2 | 0.28 | 3.2 |
| C2 | 36 | 54 | 10 | - | 2 | - | 0.2 | 0.28 | 3.2 |
| C3 | 36 | 54 | 10 | - | 3 | - | 0.2 | 0.28 | 3.2 |
| C4 | 36 | 54 | 10 | - | 4 | - | 0.2 | 0.28 | 3.2 |
| C5 | 36 | 54 | 10 | - | 5 | - | 0.2 | 0.28 | 3.2 |
| Si1 | 36 | 54 | 10 | - | - | 1 | 0.2 | 0.28 | 3.2 |
| Si2 | 36 | 54 | 10 | - | - | 2 | 0.2 | 0.28 | 3.2 |
| Si3 | 36 | 54 | 10 | - | - | 3 | 0.2 | 0.28 | 3.2 |
| Si4 | 36 | 54 | 10 | - | - | 4 | 0.2 | 0.28 | 3.2 |
| Si5 | 36 | 54 | 10 | - | - | 5 | 0.2 | 0.28 | 3.2 |

Table 5. Mixing proportions of the blended pastes (wt.%).

2.3. Testing Methods

2.3.1. Dry Shrinkage and Interconnected Porosity

Dry shrinkage test was carried out according to Chinese JTGE30-2005 Standard [37], the concrete specimen was removed from the standard curing room at 3 d age and put into a constant temperature and humidity room (temperature of 20 ± 2 °C, relative humidity of $60\% \pm 5\%$) to measure the initial length (including probe). After the initial measurement, the length of the specimen was measured at 1, 3, 7, 14, 28 and 60 d to calculate its dry shrinkage. The interconnected porosity of the concrete specimen is calculated by the mass increment of water-saturated specimen against its drying mass:

2.3.2. Abrasion Performance

According to Chinese JTGE30-2005 Standard [37], the abrasion performance of concrete specimens was tested by the underwater steel ball method. After 72 h of grinding by steel ball under water, the 28 d abrasion loss and abrasion resistance of concrete specimens were calculated as follows:

$$G_c = \frac{m_1 - m_2}{0.0125} \tag{1}$$

$$G_e = \frac{T \cdot A}{m_1 - m_2} \tag{2}$$

where G_c is 28 d abrasion loss (kg/m²), G_e is abrasion resistance (h/(kg/m²)), m_1 is initial specimen mass (kg), m_2 is specimen mass after grinding (kg), T is total grinding time and A is the abrasion area of the specimen.

2.3.3. Freeze-Thaw Test

The frost resistance of concrete was tested according to Chinese GB/T50082-2009 Standard [38]. The concrete samples were immersed in 20 ± 2 °C water for 4 d at the age of 24 d, and then the freeze–thaw circulation was taken. The freezing temperature ranges from -20 °C to -18 °C. The freezing duration is 4 h and the thawing duration is 4 h.

2.3.4. Microstructure Tests

The paste samples were prepared for microstructure tests. After reaching a specific age, the hydration of the paste was terminated with anhydrous ethanol for 24 h, and then the samples were dried in the oven at 60 °C for 24 h. X-ray diffraction (XRD) analysis was conducted using an Empyrean X-ray diffractometer, manufactured by Panaco in the Netherlands. The scanning range was 5°~50° and the step size was 4°/min. Scanning electronic microscopy (SEM) test was performed by JSM-7500F field emission scanning electron microscope, which was produced by Japan Electronics Company. Fourier transform infrared spectrometer (FTIR) was tested using Nicolet6700 made by ThermoFisher Scientific, (Waltham, MA, USA). The test was conducted in the middle infrared region with a spectral range of 4000~400 cm⁻¹ and a resolution of 2 cm⁻¹.

3. Results and Discussion

3.1. Effect of Activators on the Strength of Blended Paste

3.1.1. Effect of Na₂SO₄ Content on the Strength

Figure 3 shows the effect of Na_2SO_4 on the compressive strength of the blended paste. As can be seen from the figure, with the content of Na_2SO_4 increasing from 1% to 5%, the compressive strength of the paste at each age first increased and then decreased. The activating effect is not obvious when the content of Na_2SO_4 is low. Since the w/b ratio of paste in the group with Na₂SO₄ was higher than that in the control group, the activating effect of a small amount of Na_2SO_4 was not obvious, and hence the strength of the paste was lower than that of the control group. With increasing Na₂SO₄ content, the reaction of GBFS and SS is activated and the strength of the hardened paste increases. When the content of Na₂SO₄ was 3%, the 28 d compressive strength reached 47.8 MPa, which was 9.4% higher than the control group. This indicates that SO_4^{2-} can effectively activate the surface of SS, and promote the dissolution of vitreous structure in SS [39]. The hydration of steel slag produces calcium hydroxide, which can activate the pozzolanic reaction of GBFS. Under the combining activation effects of SS and SO_4^{2-} , slag can participate in hydration and react with CH to form ettringite and C-A-S-H gel. Ettringite and C-A-S-H gel intertwines with each other, rendering the paste structure more compact and ensuring the steady growth of its mechanical strength. When the content of Na_2SO_4 exceeds 3%, the activation effect of Na_2SO_4 is too strong, which can accelerate the setting speed of the paste. This makes the paste hard to compact during cast, resulting in a decrease in the strength of the paste. Therefore, the optimal dosage of Na_2SO_4 was determined to be 3%. It should be noted that when the dosage of Na_2SO_4 was 3%, although the 7 d and 28 d compressive strength of the paste was higher than that of the control group, the 3 d compressive strength of the paste was still lower than the control group. This indicates that Na_2SO_4 as an activator shows limited promotion on the early strength development of the paste.

3.1.2. Effect of Na₂CO₃ Content on the Strength

Figure 4 shows the effect of Na₂CO₃ activation on the compressive strength of composite cementitious paste. It can be seen that with the content of Na₂CO₃ increasing from 1% to 5%, the compressive strength of the paste first increase and then decrease for all ages. When the content of Na₂CO₃ was 4%, the 28 d compressive strength reached 44.6 MPa, which was 2.1% higher than the control group. This is because the incorporation of Na₂CO₃ can provide CO_3^{2-} ions, which can combine with calcium ions in pore solution to form a large number of calcite and aragonite minerals [40]. This can promote the refinement of the pore structure and densification of the paste. When the content of Na₂CO₃ is too high, however, CaCO₃ crystals can generate fast and agglomerate, resulting in the rapid setting of cementitious paste. This is easy to form harmful large pores in the hardened paste and is negative to its strength development. Therefore, the still increase of Na₂CO₃ content leads to decreasing strength of the composite paste. It should be noted that at the optimal addition of Na₂CO₃, the performance improvement of the activated paste is also negligible





Figure 3. Influence of Na_2SO_4 content on the compressive strength.



Figure 4. Influence of Na₂CO₃ content on the compressive strength.

3.1.3. Effect of Na₂SiO₃ Content on the Strength

Figure 5 shows the effect of Na_2SiO_3 addition on the mechanical strength of the blended paste. As shown in the figure, the compressive strength of the paste first increases and then decreases, as the content of Na_2SiO_3 increases from 1% to 5%, regardless of age. At the content of 3%, the 28 d compressive strength of the paste reaches the highest value of 50.6 MPa, which is 15.8% higher than the control group. Compared with Na_2SO_4 and Na_2CO_3 activators, Na_2SiO_3 further improves the strength of hardened paste. This implies that Na_2SiO_3 is more suitable for activating the reaction of GBFS-SS composite materials. This is because the SiO_3^{2-} ions released from Na_2SiO_3 not only can improve the alkalinity

of the pore solution to depolymerize aluminosilicate skeleton in GBFS and SS, but also can participate in the generation of C-A-S-H gel [41]. The hydration of SS can produce calcium hydroxide, which can promote the pozzolanic reaction of GBFS and react with SiO_3^{2-} and Al^{3+} ions to form more C-A-S-H gel. Therefore, the synergistic effect of the Na₂SiO₃ activation and SS reaction can improve the compactness of the hardened paste. When the content of Na₂SiO₃ exceeds 3%, the alkalinity of the paste is too high and the hardening speed is too fast, which is not conducive to the densification of the paste. Therefore, further addition of Na₂SiO₃ would result in decreasing strength of the blended paste.





3.1.4. Effect of Na₂SO₄ and Na₂SiO₃ Combination on the Strength

The above experiments show that both Na_2SO_4 and Na_2SiO_3 have a good activating effect on the GBFS-SS composite paste. Hence, this section studies the combination of Na_2SO_4 and Na_2SiO_3 on the compressive strength of the composite paste. At the basis of $3\% Na_2SiO_3$ addition, 0.5%, 1.0%, 1.5%, 2.0% and 2.5% of Na_2SO_4 were further added into the paste, respectively, in order to study the effect of combined activation. Detailed mixing proportion is given in Table 6.

| No. | SS | GBFS | Cement | Na_2SiO_3 | Na_2SO_4 | Borax |
|---------|----|------|--------|-------------|------------|-------|
| Si3S0.5 | 36 | 54 | 10 | 3 | 0.5 | 0.2 |
| Si3S1 | 36 | 54 | 10 | 3 | 1.0 | 0.2 |
| Si3S1.5 | 36 | 54 | 10 | 3 | 1.5 | 0.2 |
| Si3S2 | 36 | 54 | 10 | 3 | 2.0 | 0.2 |
| Si3S2.5 | 36 | 54 | 10 | 3 | 2.5 | 0.2 |

Table 6. Combined activation of Na₂SiO₃ and Na₂SO₄ (wt.%).

Figure 6 shows the effect of Na_2SiO_3 and Na_2SO_4 combined activation on the compressive strength of the composite blended paste. It can be seen from the figure that, as the content of Na_2SO_4 increases from 0% to 2.5%, the compressive strength of the paste at all ages increases first and then decreases. When the content of Na_2SO_4 is 1%, the compressive strength reaches the highest value, meaning the optimal incorporating content is 3% for Na_2SiO_3 and 1% for Na_2SO_4 . The 7 d and 28 d compressive strength of the Si3S1 group paste increased by 1.6% and 1.5% compared to the control group, respectively. This

indicates that the combined activation of Na_2SiO_3 and Na_2SO_4 can further promote the hydration of GBFS-SS paste and, hence, improve its compressive strength [42,43]. It can be seen that the 3 d compressive strength of the Si3S1 group paste is evidently reduced compared with the control group, indicating that the addition of Na_2SO_4 would retard the early strength development of the paste [44]. This is the same as the law when in a single Na_2SO_4 -activated sample.



Figure 6. Influence of Na₂SO₄-Na₂SiO₃ combination on the compressive strength.

3.2. Effect of Na_2SO_4 and Na_2SiO_3 Combination on the Microstructure of GBFS-SS Composite Paste

3.2.1. X-ray Diffraction

Figure 7 shows the XRD patterns of the 3 d and 28 d aged composite pastes activated with both Na₂SO₄ and Na₂SiO₃. As shown in the figure, under the combined activation of Na₂SO₄ and Na₂SiO₃, the main hydration products of the composite pastes are ettringite (AFt), portlandite (CH) and amorphous C-A-S-H gel. Additionally, a small amount of zoisite was also produced. However, the inert mineral components in SS, such as the RO phase and C_2F , are reacted very slowly. The relative intensities of their diffraction peaks remain unchanged [45]. With the extension of hydration age, the relative strength of diffraction peaks of C₂S and C₃S gradually decreases, while that of portlandite gradually increases, indicating that portlandite and C-S-H gel are produced during the hydration. As compared with the control group, the relative strength of ettringite diffraction peaks increased significantly after the addition of Na₂SO₄ and Na₂SiO₃, because the addition of both provides a large amount of SO_4^{2-} and SiO_3^{2-} in the system. Under the synergic activation of SiO_3^{2-} and SO_4^{2-} , the surface of slag particles dissolved and depolymerization occurred. Active silicate tetrahedra [SiO₄] and aluminate tetrahedra [AlO₄] were formed, which further promoted the production of C-A-S-H gel and ettringite. Moreover, the relative strength of ettringite diffraction peaks increases with the extension of hydration age, indicating that ettringite is continuously generated in the paste. Meanwhile, a large number of silicates hydrolyzed to produce H_3SiO^{4-} and OH^- , which could maintain the alkalinity of the paste and promote the continuous polymerization of silicate tetrahedra.



Figure 7. XRD patterns of the composite paste with Na₂SO₄ and Na₂SiO₃ combined activation; (a) 3 d; (b) 28 d.

3.2.2. Fourier Transform Infrared

Figure 8 shows the FTIR spectra of the combined activated GBFS-SS composite pastes at a hydration age of 3 d and 28 d. Among them, the sharp peak around 3644 cm^{-1} is the stretching vibration peak of the free OH bond, indicating that a small amount of portlandite is generated after 3 d of hydration. This is because under the activation of SO_4^{2-} , the SS hydrated produces portlandite. As the amount of Na₂SO₄ increases, more hydration products had been generated in the system, and these hydration products would react with portlandite to form C-A-S-H gel. Around 3443 cm⁻¹ and 1642 cm⁻¹ are the stretching and bending vibration peaks of the H-O-H bond of the bonded water in hydration products, respectively, indicating that a large number of amorphous C-A-S-H gel are generated in the paste. With an increased amount of Na₂SO₄ or the extension of hydration, the corresponding transmitted waves gradually shift towards the high wave number. This indicates that the degree of polymerization of hydration products increases gradually. However, 1425 cm⁻¹ and 875 cm⁻¹ are caused by the asymmetric stretching vibration and buckling vibration of CO_3^{2-} , respectively, which may be due to the carbonation of f-CaO in SS or portlandite [46]. The band near 1108 cm^{-1} and 618 cm^{-1} is the vibration band of SO_4^{2-} in ettringite. With the higher content of Na₂SO₄, the SO_4^{2-} vibration band becomes more pronounced, indicating an increasing amount of ettringite generation.



Figure 8. FTIR spectra of GBFS-SS composite pastes activated with Na₂SiO₃ and Na₂SO₄; (**a**) 3 d; (**b**) 28 d.

3.2.3. Scanning Electronic Microscopy

Figure 9 shows the 3 d, 7 d and 28 d micrographs of hydrated GBFS-SS composite paste with 3% Na₂SiO₃ and 1% Na₂SO₄ activation. As can be seen from the figure, when the hydration age reaches 3 d, a large number of C-A-S-H gel-like fiber networks and a small amount of hexagonal-shaped portlandite sheet have been generated in the system. At this age, the sample structure is loose, with a higher proportion of pores, and the hydration products are isolated. With the progression of hydration, the amount of hydration products increases and their crystallinity is gradually enhanced. The fibrous C-A-S-H gel gradually developed into clusters and intertwined with ettringite and portlandite to form a threedimensional network structure, refining the pore structure and improving the compactness of the paste.



(a)

(b)

Figure 9. The SEM images of GBFS-SS composite paste activated with 3% Na₂SiO₃ and 1% Na₂SO₄; (a) 3 d; (b) 7 d; (c) 28 d.

3.3. The Performance of GBFS-SS Composite Pavement Concrete

Based on the investigation of the strength and microstructure of GBFS-SS composite pastes, this section studies the influence of different activators on the performance of composite materials concrete. The w/b ratio of the concrete sample with activators is set to 0.3 and the sand ratio is set at 0.43. The water-binder ratio and sand rate of the concrete in the control group was set as 0.29 and 0.42. The mixing proportion of concrete was shown in Table 7, where the addition of superplasticizer was modified to make the concrete have proper workability.

| fable 7. Mix | ing proportion | of GBFS-SS c | composite pavem | ent concrete | (kg/m ³ | ³). |
|---------------------|----------------|--------------|-----------------|--------------|--------------------|-----------------|
|---------------------|----------------|--------------|-----------------|--------------|--------------------|-----------------|

| No. | GBFS | SS | Cement | Basalt Stone | Sand | Water | Superplasticizer /% | Borax /% | Activators and Content/% |
|----------------|------|-----|--------|-----------------|------|-------|------------------------|-------------|---|
| Cc S3c | 324 | 216 | 60 | 956 | 692 | 156 | 3.6 2.4 | - | - 3% Na ₂ SO ₄ |
| Si3c Si3S1c | 324 | 216 | 60 | 940 | 708 | 162 | 3.2 3.8 | 0.2 | 3% Na ₂ SiO ₃ 3% Na ₂ SiO ₃ +1% Na ₂ SO ₄ |

3.3.1. Workability and Compressive Strength

Figures 10 and 11 show the workability and mechanical strength of concrete samples. It can be observed that the slump is higher than 200 mm and the slump flow is between 500–600 mm for each concrete group, showing their good workability. The 3 d, 28 d and 60 d compressive strengths of GBFS-SS pavement concrete activated with Na₂SO₄ are 34.3 MPa, 59.2 MPa and 69.4 MPa, respectively. The early compressive strengths are lower than those of the control group, but the 28 d and 60 d compressive strengths are increased by 2.4% and 4.8%, respectively. Under the activation of Na₂SiO₃, the compressive strength of GBFS-SS pavement concrete at 3 d, 28 d and 60 d increased by 6.1%, 4.5% and 7.7% compared to the control group, respectively. The 3 d, 28 d and 60 d compressive strengths

of concrete with combined activation of Na_2SiO_3 and Na_2SO_4 are 46.3 MPa, 61.7 MPa and 73.5 MPa, respectively, showing the highest mechanical strength. The results show that the combined activation has benefit to the strength development of concrete than the activation of Na_2SiO_3 or Na_2SO_4 alone.



Figure 10. Influence of activators on the workability of concrete.



Figure 11. Influence of activators on the mechanical strength of concrete.

3.3.2. Dry Shrinkage

Figure 12 shows the drying shrinkage of composite pavement concrete mixed with different activators. It can be seen from the figure that the shrinkage of the control group was 25×10^{-6} at 1 d. The shrinkage of concrete activated with different activators was reduced compared with the control group. Under the activation of Na₂SO₄ and Na₂SiO₃, the early drying shrinkage decreased by 90% and 80%, respectively. This indicates Na₂SO₄ and Na₂SiO₃ can both reduce the dry shrinkage of the composite concrete. The reason for Na₂SO₄ addition reducing early shrinkage reduction of concrete is due to the compensated shrinkage effect of ettringite formation, while Na₂SiO₃ addition can reduce shrinkage of composite concrete due to the rapid development of its early strength and the enhancing resistance to deformation. The combined activation of Na₂SiO₃ and Na₂SiO₄ reduced the early drying shrinkage by 86%, which shows a moderate effect in reducing the dry

shrinkage. At the age of 60 d, the shrinkage of the control group concrete was 240×10^{-6} , while that for the Na₂SO₄ activated group was 220×10^{-6} . The shrinkage of the Na₂SiO₃-activated group was 285×10^{-6} , which is 18.75% higher than the control group. This shows that Na₂SO₄ activation can continuously reduce the dry shrinkage of the concrete, whereas Na₂SiO₃ would enlarge the shrinkage of the concrete sample at an elderly age. The reason for the evident shrinkage in Na₂SiO₃ activated group at the later stage may be caused by its alkali activation [47]. The volume of mesopore (<50 nm) in the alkaline activated materials was about one time higher than the ordinary Portland cement paste, resulting in their significant capillary pore shrinkage. With respect to Na₂SO₄ activation, it can compensate for the shrinkage of the Na₂SiO₃ and Na₂SO₄ combined activation group is 270×10^{-6} , with an increase of 12.5% compared to the control group. This indicates that under Na₂SO₄ and Na₂SiO₃ combination, the formation of ettringite promoted by Na₂SO₄ can still play a role in shrinkage compensation, and the problem of later-stage shrinkage caused by Na₂SiO₃ activator can be effectively moderated.



Figure 12. Influence of activators on the dry shrinkage of pavement concrete.

The shrinkage of each concrete group hydrated for 60 d is still lower than 300 $\mu\epsilon$, which meets the requirement of pavement concrete. This could be attributed to the internal curing effect of high titanium heavy slag sand [48]. In the concrete sample with Na₂SO₄ addition, the internal curing water can promote the formation and expansion of ettringite, which can further reduce the volume shrinkage.

3.3.3. Interconnected Porosity

Figure 13 shows the interconnected porosity of concrete with different activators addition. It can be seen that, for all groups of samples, the decrease of interconnected porosity of concrete from 3 d to 28 d is significantly greater than that from 28 d to 60 d, the decrease of the former is 26.76%, while the decrease of the latter is only 7.69%. This is because the hydration of the concrete is faster in the early stage. The pores are quickly filled with hydration products. The hydration degree of the late-age system is high. Most of the connected pores in the hardened paste have been filled, and the pore structure has been optimized and improved. It is difficult to further substantially refine the pores, so the porosity reduction is minor. Compared with the control group, when salt or alkali activators are added, the interconnected porosity of concrete at each age is significantly

reduced. This is because the hydration degree of the composite cementitious system will be further improved with the extension of the age, and the generated hydration products continue to fill the connected pores in the concrete, so the interconnected porosity gradually decreases. The variation of the interconnected porosity of concrete with different activators is negatively correlated with its compressive strength; that is, the higher the compressive strength of concrete, the lower the interconnected porosity. This is because the addition of an activator promotes hydration of the cementitious materials, which forms more amount of hydration products, and improves the compressive strength of concrete when the hydration products fill the capillary pores. Therefore, increasing mechanical strength is also accompanied by the reduction of interconnected porosity.



Figure 13. Influence of activators on the interconnected porosity of pavement concrete.

3.3.4. Abrasion Performance

Figure 14 shows the abrasion loss and abrasion resistance of concrete activated with different activators. As shown in the figure, the abrasion resistance of composite pavement concrete activated by Na₂SO₄ and Na₂SiO₃ has been improved by 15.2% and 22.4% compared to the control group concrete, respectively. Accordingly, the abrasion loss of Na₂SO₄ and Na₂SiO₃-activated concrete samples is 13.1% and 18.3% lower than the reference group, respectively. The combined activation of Na₂SiO₃ and Na₂SO₄ shows the most obvious effect on the abrasion performance of concrete. The abrasion resistance of concrete is $28.32 \text{ h}/(\text{kg/m}^2)$, which is 32.9% higher than that of the control group. The abrasion loss is 2.542 kg/m^2 , which is 24.7% lower than the control group. There is a good correlation between the abrasion resistance test results and the compressive strength test results of concrete. This implies that the combined activation of Na₂SiO₃ and Na₂SiO₃ and Na₂SO₄ effectively promotes the hydration of paste, and more hydration products are formed to make concrete denser, so its wear resistance is greatly increased.



Figure 14. Influence of activators on the abrasion performance of pavement concrete.

3.3.5. Freeze-Thaw Resistance

Table 8 shows the mass loss and compressive strength loss of concrete with different activators under different freeze-thaw cycles. As can be seen from the table, the freeze-thaw resistance of concrete is greatly improved as the Na₂SO₄ is added. Its mass loss ratio and strength loss ratio of the concrete sample after 100 times of freeze-thaw cycles is 0.80% and 9.51%, respectively. As compared with the control group, the mass loss and strength loss decreased by 20.8% and 19.9%, respectively. The mass loss and strength loss of the concrete activated by Na₂SiO₃ were 0.84% and 9.38% after 100 freeze-thaw cycles, respectively, which decreased by 16.8% and 21.0%, compared with the control group. The combined activation of Na₂SiO₃ and Na₂SO₄ shows the best freezing-thawing resistance. The mass loss and strength loss for the combined activated sample are 0.73% and 8.74%, respectively. As compared with the other sample, the combined activated sample has the lowest mass loss and strength loss after freeze-thaw cycling. The freeze-thaw resistance of concrete samples is consistent with their compressive strength. This is because the failure of GBFS-SS composite pavement concrete under freeze-thaw cycle is originated from the expansive crystallization stress of water in the pore solution. In the freezing stage, micro-cracks occur due to the crystallization stress of the pore solution, and in the melting stage, more water is absorbed through micro-cracks. Subsequently, more ice crystals are produced in the next freezing stage, resulting in further increasing crystallization stress. Therefore, reducing the initial porosity of concrete can improve its freeze-thaw resistance. The combined activation of Na₂SiO₃ and Na₂SO₄ can promote the hydration of composite paste. This causes more hydration product formation and filling of the capillary pores. Therefore, the freeze-thaw resistance of the pavement concrete is improved as its compressive strength increases.

Table 8. The freeze-thaw cycling resistance of concrete with different activators.

| No. | Ν | /lass Loss Ratio/ | % | Comp | Classification | | |
|-------|----------|-------------------|-----------|----------|----------------|-----------|-------|
| | 25 Times | 50 Times | 100 Times | 25 Times | 50 Times | 100 Times | |
| Cc | 0.52 | 0.71 | 1.01 | 5.24 | 8.01 | 11.87 | >D100 |
| S3 | 0.37 | 0.54 | 0.80 | 3.86 | 6.12 | 9.51 | >D100 |
| Si3 | 0.38 | 0.50 | 0.84 | 3.72 | 6.26 | 9.38 | >D100 |
| Si3S1 | 0.34 | 0.47 | 0.73 | 3.36 | 5.92 | 8.74 | >D100 |

4. Conclusions

This work prepared GBFS-SS composite concrete with different activators. The effect of activators on the compressive strength of GBFS-SS composite paste was studied to obtain their optimal content. Then, the mechanical properties, dry shrinkage, interconnected porosity, abrasion resistance and freeze–thaw resistance of GBFS-SS composite concrete were studied. Microscopic testing techniques of XRD, FTIR and SEM were implemented to elucidate the activation mechanisms. Conclusions can be made as follows:

- (1) Both Na₂SO₄ and Na₂SiO₃ can promote the strength development of the GBFS-SS composite. The composite paste with 3% of Na₂SO₄ addition has a 28 d compressive strength of 47.8 MPa, which is 9.4% higher than the control group. The composite paste with 3% of Na₂SiO₃ addition has a compressive strength of 50.6 MPa, which is 15.8% higher than the control group. Na₂CO₃ shows a limited effect on the strength development of GBFS-SS composite paste. At the optimal Na₂CO₃ content of 4%, the 28 d compressive strength of the composite paste is only 44.6 MPa, which is 2.1% higher than the control group.
- (2) As compared with Na₂SiO₃ or Na₂SO₄ single activation, the combined activation of Na₂SiO₃ and Na₂SO₄ shows a better improving effect on the strength development of the composite paste. The GBFS-SS composite concrete with 3% of Na₂SiO₃ and 1% of Na₂SO₄ addition has a 60 d compressive strength of 73.5 MPa, a 60 d drying shrinkage of 270×10^{-6} , a 60 d interconnected porosity of 6.85%, and the 28 d abrasion resistance of 28.32 h/kg/m².
- (3) All the GBFS-SS composite paste contains the hydration products of ettringite, portlandite and amorphous C-A-S-H gel. The Na₂SiO₃ and Na₂SO₄ activated paste has an extra hydration product of zoisite. SO_4^{2-} can accelerate the depolymerization of the aluminosilicate network in GBFS and SS. SiO_3^{2-} not only facilitates the pozzolanic reaction of GBFS and SS, but also participates in the hydration to form more C-A-S-H gel.

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