Road performance analysis of cement stabilized coal gangue mixture

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Abstract

In order to solve the environmental pollution of coal gangue and the shortage of aggregate resources in road engineering, waste coal gangue is used as road base material instead of natural stone materials. Through physical, mechanical, chemical and activity tests of coal gangue aggregate, the optimal gradation composition of unconfined compressive strength was determined. Through unconfined compressive strength, indirect tensile strength, flexural tensile strength, freeze-thaw and dry shrinkage tests, the influence of cement content on road performance of cement stabilized coal gangue mixture was studied. By means of SEM, ICP AES, XRD and optical digital microscope, the difference between spontaneous combustion coal gangue and Unspontaneous combustion coal gangue was analyzed, the microstructure of cement stabilized coal gangue mixture was characterized, and the strength formation mechanism of mixture was explored. The results show that Spontaneous combustion coal gangue has higher activity than Unspontaneous combustion coal gangue. Based on the selected optimal allocation (BNS:SNS:SSC = 71.26:9.41:18.8). The mixture of 4% cement dosage can not only meet the requirement of early strength 4.16 MPa, but also show an efficient strength growth rate of 36.10%, showing the optimum mechanical properties. The total shrinkage coefficient of cement stabilized coal gangue mixture with 4% cement dosage is $1.12 \times 10^{-2}$, which shows that the dry shrinkage resistance is the best. With the increase of time, hydration degree is gradually deepened, and gelled substance is more tightly bonded to aggregates. There is no obvious gap between aggregates, and the integrity of the mixture is enhanced, which can show better road performance. Ca(OH)$_2$, a cement hydration product in cement stabilized coal gangue mixture, takes place pozzolana reaction with active SiO$_2$ and Al$_2$O$_3$ in coal gangue to produce gismondine, which is beneficial to the global strength and the bond quality of the mixture.

1. Introduction

Coal gangue is a gray solid waste produced in the process of coal excavation and production. Coal gangue is associated with coal in the process of coal carbon formation. Coal gangue produced in the process of coal mining accounts for about 10% ~ 25% of the total [1]. With the increase of coal mining volume year by year, the coal gangue produced by coal mining can’t be rationally used, resulting in the increase of coal gangue storage volume year by year, and the problems of land occupation and pollution can’t be ignored [2]. Self-combustion waste rock hill will also cause explosion, because news of people injured by coal gangue pile explosion is common...
Cement stabilized coal gangue is maintained at about 90%. The increase of cement dosage is beneficial to improve the frost resistance index can meet the requirements [4]. After five freeze-thaw cycles, the strength guarantee rate of cement stabilized coal gangue is maintained at about 90%. The increase of cement dosage is beneficial to improve the frost resistance [5]. Compared with the research on the road performance of cement stabilized coal gangue mixture, the research on the road performance of cement stabilized macadam mixture is more extensive and in-depth. There is a correlation between the compressive strength and the indirect tensile strength of cement stabilized macadam mixture with higher cement dosage is low [7]. By adding cement to lime-fly ash stabilized coal gangue mixture, the freeze-thaw resistance of lime-fly ash stabilized coal gangue mixture can be improved, and the minimum cement content can meet the requirements of freeze-thaw cycle [8]. Reactive SiO2 and Al2O3 in thermal-activated coal gangue react with Ca(OH)2 to form calcium silicate hydrate and calcium aluminous hydrate gel to improve the compressive strength of cement [9]. The application of waste gypsum modified total waste four slag mixture pavement base is completely feasible. Not only stable road performance, high early strength, but also make full use of industrial waste, reduce costs, avoid environmental pollution [10]. Using coal gangue as production materials of gelled substance not only reduces production cost, but also helps to consume coal gangue waste. Organic matters extracted from coal gangue waste can be used for the production of agrochemical raw materials, and unusable coal gangue waste can be used for backfilling soil after treatment [11]. Unlike European and American countries, Eastern European countries mainly use coal gangue waste to produce construction materials. Coal gangue as main raw material can be used to produce bricks and road materials [12].

Secondary Utilization of Industrial Solid Waste Resources by Coal Gangue Waste in Eastern European Countries [13]. When the blended cement paste contains 5% coal gangue and 25% mineral slag, the drying shrinkage of the composite is smaller and the compressive strength is larger [14]. The smaller the proportion of activated coal gangue instead of cement, the better the drying shrinkage. When activated coal gangue replaces 50% cement, the temperature shrinkage coefficient of cement stabilized macadam base is the least in unfavorable temperature range. The optimum substitution amount of calcined coal gangue for cement in cement stabilized macadam base is 40%~50% [15]. The active components SiO2 and Al2O3 of coal gangue fine aggregate can react with cement hydration products secondary hydration reaction to a certain extent. Compared with early age cement mortar, it can further improve the microstructure of mortar, enhance the interface performance of mortar, make the internal structure of mortar more uniform, and increase the late strength of cement hardened mortar greatly. The pozzolan effect of coal gangue fine aggregate calcined at 700 °C with the highest activity is obvious [16]. Shrinkage performance of materials with large particle size is small, and shrinkage performance of fine aggregate such as powder is obvious [17]. When the cement strength is the same, the drying shrinkage increases with the increase of water cement ratio. When the concrete strength is similar, the drying shrinkage decreases with the increase of cement strength. The strength of full recycled aggregate concrete decreases and shrinkage performance increases without prewetting treatment [18]. The optimal water consumption increases with the increase of cementitious material dosage and sand ratio. The optimal water glue ratio decreases with the increase of the amount of gelled substance, and decreases with the increase of age [19]. Inhibition of CO2 formation between coal gangue and biomass has positive significance for greenhouse gas emission reduction [20]. Due to the high moisture content of coal gangue particles, the fracture toughness and fracture energy of concrete are significantly reduced by freeze-thaw cycles. There is a good correlation between freeze-thaw times and fracture toughness and fracture energy damage of coal gangue concrete after freeze-thaw cycles [21]. Coal gangue powder—mineral powder 1:1 as filler, 0.4% polyester fiber dosage, asphalt mixture can form high viscosity dense thick asphalt film and fiber formed three-dimensional network structure can well alleviate and reduce the damage of salt freezing erosion on the mixture [22].

In summary, although the comprehensive utilization of coal gangue has been studied in China, and the road performance, frost resistance and drying shrinkage performance of cement stabilized coal gangue mixture base are studied, there is no corresponding technical specification or standard at present. In addition, the activity of coal gangue activity, phase analysis and microstructure characterization of cement stabilized coal gangue mixture are less, and the performance of coal gangue will also vary with different origins. The research on the
characteristics of coal gangue produced by coal mines in various origins is not enough. At present, coal gangue can only be applied to low grade road subgrade and pavement subbase, which seriously limits the large-scale application of coal gangue. Based on the road performance of cement stabilized coal gangue mixture base, this paper explores the mechanical development law of cement stabilized coal gangue mixture base, analyzes the strength formation mechanism of cement stabilized coal gangue mixture base, and provides theoretical support for the application of cement stabilized coal gangue mixture base to high grade paving direction research.

2. Raw material

2.1. Cement

The cement used in the test is Xiujian P.O 42.5 cement produced by Baiquan, Huixian City. In order to test the performances of cement, the cement fineness, setting time and strength of cement mortar are tested. The test results are shown in Table 1.

| Cement grade | Fineness (%) | Standard consistence (%) | Initial setting time (min) | Final setting time (min) | 3d/28d Strength of cement mortar (MPa) |
|--------------|--------------|--------------------------|---------------------------|-------------------------|---------------------------------------|
| P.O 42.5     | 4.12         | 28.5                     | 215                       | 525                     | 4.8/7.5                               |
| Standards    | ≤10          | 22–32                    | ≥180                      | 360~600                 | ≥3.5/6.5                              |

It can be seen from Table 1 that the cement fineness used in the test is 4.12%, the normal consistence is 28.5%, the initial setting time is 215 min, and the final setting time is 525 min. The anti-fracture and compressive strength of cement gel sand for three days are 4.8 MPa and 24.5 MPa respectively. The anti-fracture and compressive strength of cement gel sand for 28 days are 7.5 MPa and 44.8 MPa respectively, which all meet the construction specifications of pavement base.

2.2. Coal gangue aggregate

2.2.1. Coal gangue crushing

The coal gangue used is produced in the mining process of Liguizuo Village, Jiaozuo City, Henan Province. The stone materials are mostly gray or gray black, and the part of coal gangue outside the coal gangue mountain is reddish brown, as shown in figure 1(a). The exposed parts of coal gangue stones are reddish brown, while the buried parts are gray black, as shown in figure 1(b). Because the rock block is large and hard, the coal gangue is broken into large size coal gangue aggregate (group A) and small size coal gangue aggregate (group B) by jaw crusher.

2.2.2. Coal gangue crushing and chemical composition

The coal gangue is crushed into powder by electromagnetic ore crusher. After crushing, the spontaneous combustion coal gangue is yellowish brown, and the unsponstaneous combustion coal gangue is gray. The following figure 2 shows.

![Coal gangue hill in Jiaozuo](image1)

![Part exposed part buried coal gangue](image2)

Figure 1. Coal gangue sampling.
Inductively coupled plasma spectrometer PerkinElmer 730 was used to quantify the main chemical components of spontaneous combustion coal gangue and unspontaneous combustion coal, such as table 2.

2.2.3. XRD patterns of coal gangue
The XRD pattern of coal gangue is shown in figure 3.

3. Test method

3.1. Mix proportion design method of cement stabilized coal gangue mixture

3.1.1. Determine gradation design
According to the recommended gradation range of C-B-1 for expressway and first-grade highway in 《Technical Rules for Highway Pavement Base Construction》 (JTG / T F20-2015) [23], the maximum particle size of coal gangue was selected as 26.5 mm, and the aggregate of group A and B was synthesized. When the aggregate of group A and group B was mixed in accordance with the ratio of 2:8, the obtained aggregate can just meet the mix proportion range of framework dense structure requirements in 《Technical Rules for Highway Pavement Base Construction》 (JTG / T F20-2015). The results are as follow: figure 4.

3.1.2. Determine mix proportion design
The hardness of spontaneous combustion coal gangue is lower than that of unspontaneous combustion coal gangue. Therefore, if the coarse aggregate of cement stabilized coal gangue mixture is replaced by spontaneous combustion coal gangue instead of unspontaneous combustion coal gangue, the compressive strength of cement stabilized coal gangue mixture will inevitably decrease. Related research shows that the influence of spontaneous combustion coal gangue on cement gel sand specimens mainly comes from the chemical reaction of active ingredients in spontaneous combustion coal gangue [24]. Therefore, this experiment determines that the proportion of spontaneous combustion coal gangue and unspontaneous combustion coal gangue affects the road performance of coal gangue mixture only considering the change of part of coal gangue in fine aggregate (less than 2.36 mm aggregate), and unspontaneous combustion coal gangue is still used in aggregate greater than 2.36 mm.

The specific test arrangement and steps are as follows: the broken coal gangue of group A and group B are evenly mixed according to the ratio of 2:8, and the vibrating screen machine is used to pass the 2.36 mm sieve hole. After screening, the mass ratio of unspontaneous combustion coal gangue aggregate (hereafter referred to as BNS) larger than 2.36 mm particle size and unspontaneous combustion coal gangue aggregate (hereafter referred to as SNS) smaller than 2.36 mm particle size should be 71.76:28.24. Spontaneous combustion coal gangue is broken into aggregate with maximum particle size no more than 2.36 mm by jaw crusher (due to the limited crushing particle size of jaw crusher and artificial crushing of large aggregate after crushing). Spontaneous combustion coal gangue aggregate (hereinafter referred to as: SSC) after screening gradation ratio curve is shown in table 3.
In order to explore the influence of spontaneous combustion coal gangue in coal gangue aggregate on the performance of cement stabilized coal gangue mixture, four groups of fine aggregate spontaneous combustion coal gangue with different proportions below 2.36 mm were designed to replace and unspontaneous combustion coal gangue mixture. As shown in table 4, the four groups of coal gangue aggregate had the same gradation, and the differences caused by different gradations were excluded. Four groups of cement stabilized coal gangue mixture cement dosage unified use of engineering commonly recommended cement dosage 4%.

3.2. Research methods of mechanical properties

3.2.1. Compaction test
Because the gradation range used in this test is the skeleton dense structure range, the vibration compaction method can make the mixture form a more effective skeleton structure than the ordinary heavy compaction method, and can more effectively simulate the working mode of the vibratory roller on the construction site. In

Figure 3. XRD patterns of coal gangue.
this paper, 3%, 4%, 5%, 6% four different cement content of indoor standard compaction test to determine the optimum moisture content of four groups of mixture and maximum dry density.

3.2.2. Unconfined compressive strength test
The unconfined compressive strength is the most simple and direct index to show the mechanical properties of materials, and it is also the main quality control project in the process of road engineering construction. In this experiment, 600 kN microcomputer controlled electro-hydraulic servo testing machine (WAW-600B) was used to test the unconfined compressive strength of the specimen. The failure process of the specimen (7 d age) is shown in figure 5.

3.2.3. Indirect tensile strength test
The specimen was formed by vibration compaction method, and the size of the specimen was diameter × height = 150 mm × 150 mm. The specimen was saturated in water one day before the test. According to ‘Indirect Tensile Strength Test Method for Inorganic Bond Stabilized Materials (Splitting Test)’ (T 0806-1994), the indirect tensile strength test of specimens was carried out by 600 kN microcomputer controlled electro-hydraulic servo testing machine (WAW-600B).

3.2.4. Flexural tensile strength test
At present, the commonly used bending-tensile test method of inorganic binder is the three-point compression method. Forming specimen by vibration compaction method, specimen size is long × wide × high = 400 mm × 100 mm × 100 mm. According to ‘inorganic binder stabilized material flexural tensile strength test

Table 3. SSC aggregate gradation ratio.

| Size of screen mesh (mm) | 2.36 | 1.18 | 0.6 | 0.3 | 0.15 | 0.075 |
|-------------------------|------|------|-----|-----|------|-------|
| By percentage (%)       | 100  | 68.70| 48.30| 29.89| 15.40| 9.03  |

Table 4. Composition of coal gangue in different groups.

| Groups               | Composition of coal gangue aggregate |
|----------------------|--------------------------------------|
| The first group      | BNS:SNS = 71.26:28.24                |
| The second group     | BNS:SNS:SSC = 71.26:18.83:9.41       |
| The third group      | BNS:SNS:SSC = 71.26:9.41:18.83       |
| The fourth group     | BNS:SSC = 71.26:28.24                |

Figure 4. Synthetic gradation curve.
method’ (T 0851-2009), 600 kN microcomputer control electro-hydraulic servo testing machine (WAW-600B) was used to test the flexural tensile strength of the specimen.

3.3. Research methods of durability

3.3.1. Freeze-thaw cycle test

In this experiment, the specimen was formed by vibration compaction method. The size of the specimen was diameter × height = 150 mm × 150 mm, and it was maintained for 180 days. According to ‘Freeze-thaw test method for inorganic binder stabilized materials’ (T 0858-2009), a total of 10 freeze-thaw cycles of 16 h of −18 °C freezing and 8 h of 20 °C melting were carried out by Automatic cryogenic freeze-thaw tester (figure 6(a)) (figure 6(b)), and the compressive strength of specimens was tested by 600 kN microcomputer control electro-hydraulic servo testing machine (WAW-600 B).

After curing for 180 days, the hydration reaction process of cement stabilized coal gangue mixture is very slow, and the strength increase and quality change caused by hydration reaction during 10-day of freeze-thaw cycle can be ignored. Figure 6(b) shows the picture of drainage at the end of 1 freeze-thaw cycle with 3% cement dosage. It can be seen from the figure that due to the less bonding agent on the surface of the specimen with low cement dosage, there are gaps between the aggregates and some aggregates fall off.

3.3.2. Dry shrinkage test

In this section, the crack resistance of cement stabilized coal gangue was analyzed through the dry shrinkage test. The specimen was formed by vibration compaction method. The size of the specimen was length × width × height = 400 mm × 100 mm × 100 mm. After seven days of immersion saturation, the specimen was dried and put into the dry shrinkage measurement mould and the dry shrinkage chamber, as shown in figure 7.

Referring to the ‘dry shrinkage test method for inorganic binder stabilized materials’ (T 0854-2009), the dry
shrinkage of the specimen was measured by a micrometer. The length shrinkage of the specimen was measured and weighed every day on the first 7 days, and the measurement test was carried out every two days from the 7th to the 29th days, and the measurement test was carried out on the 60th and 90th days.

3.4. Micro-analysis

3.4.1. Coal gangue activity analysis

At present, there are many methods for evaluating the activity of volcanic ash, and ICP AES is a fast and effective method for analyzing the activity of volcanic ash. In this experiment, this method was used to evaluate the activity content of SiO$_2$ and Al$_2$O$_3$ in coal gangue.

The test process and steps are as follows: After the crushed spontaneous combustion coal gangue and unspontaneous combustion coal gangue were screened by 100 mesh, 1g of them were dissolved in 100 ml 1 mol l$^{-1}$ Na(OH)$_2$ solution by analytical balance, and the mixed solution was placed at room temperature ($20 ^\circ C \sim 25 ^\circ C$) for 7 days. The dissolution of Si$^{4+}$ and Al$^{3+}$ was tested by inductively coupled plasma spectrometer.

3.4.2. XRD test

In order to study the influence of active components in coal gangue on the strength of cement stabilized coal gangue mixture, based on the composition of coal gangue aggregate in cement stabilized coal gangue selected in the previous experiment as BNS:SNS:SSC = 71.26:9.41:18.83, the phase analysis of 7-day and 180-day cement stabilized coal gangue mixture was carried out by PANalytical (Panaco) Xpert$^{\text{TM}}$ Powder X-ray diffractometer.

3.4.3. Scanning electron microscope test

Zeiss EVO HD15 scanning electron microscope was used to observe the cement stabilized coal gangue mixture for 7 d and 180 d. The microscopic morphology of cement stabilized coal gangue under different cement dosages at the same age was compared and analyzed, and the strength formation mechanism of cement stabilized coal gangue mixture was analyzed.

4. Results and analysis

4.1. Mechanical properties analysis

4.1.1. Compaction test

The optimum moisture content and maximum dry density of cement stabilized coal gangue mixture with different cement dosages are shown in table 5.

![Figure 7. Dry shrinkage test of cement stabilized gangue mixture.](image)

Table 5. Vibration compaction test data of cement stabilized coal gangue mixture.

| Groups | Cement dosage (%) | Maximum dry density (g/cm$^3$) | Optimum moisture content (%) |
|--------|-------------------|-------------------------------|-----------------------------|
| 1      | 3                 | 2.30                          | 6.3                         |
| 2      | 4                 | 2.31                          | 6.5                         |
| 3      | 5                 | 2.33                          | 6.7                         |
| 4      | 6                 | 2.32                          | 7.1                         |

It can be seen from table 5 that the optimum moisture content increases with the increase of cement dosage, and the two are positively correlated. The greater the cement dosage, the greater the water requirement, and the corresponding optimum moisture content increases; the relationship between the maximum dry density and
cement dosage is a hump-shaped curve that climbs to the top and then decreases, because the increase of water volume can play the role of bonding aggregate to increase the compactness, but when the proportion of water continues to increase, the proportion of corresponding aggregate in the unit volume after compaction decreases, so the compactness decreases.

4.1.2. Unconfined compressive strength

The trend chart is drawn between the cement dosage at 7 days and the unconfined compressive strength of the cement stabilized coal gangue mixture. As shown in figure 8, the relationship between the cement dosage of the cement stabilized coal gangue mixture and the unconfined compressive strength at 7 days is a cubic polynomial curve relationship that is steep first and then slow, and the influence of the increase of cement dosage on the compressive strength of the cement stabilized coal gangue is gradually weakened.

The strength of cement stabilized coal gangue mixture is mainly composed of cement hydration products and coal gangue skeleton structure. During the cement dosage of 3% to 5%, the cement hydration products increase relatively with the increase of cement dosage, which makes the combination between coal gangue aggregates better and the corresponding mixture strength increase. However, when the cement dosage continues to increase, the specific surface area of the mixture increases, the water consumption increases, and the compaction degree of the mixture is affected. Moreover, due to the increase in the proportion of fine aggregate in the mixture, the effective skeleton structure formed by coal gangue coarse aggregate in the mixture is relatively reduced, so the increase in the strength of the mixture is slowed down.

The compressive strength of cement stabilized coal gangue mixture with 3%, 4%, 5% and 6% cement ratio at each age is drawn into a trend chart. Figure 9 shows that the cement stabilized coal gangue mixture has high early strength, and the age of the mixture specimen is positively correlated with the compressive strength. Due to the early hydration reaction of tricalcium silicate in cement, the strength of cement stabilized coal gangue mixture increased rapidly from 7 days to 14 days. The strength of cement stabilized coal gangue with 3% cement dosage increased by about 28.83%, and the strength of cement stabilized coal gangue mixture with 4%, 5% and 6% cement dosage increased by about 1.5 MPa, with growth rates of 36.10%, 30.28% and 32.42%, respectively. With the development of time, the cement hydration slowed down, the reaction between active ingredients in spontaneous combustion coal gangue and cement hydration products gradually stopped, and the growth rate of unconfined compressive strength of the mixture gradually slowed down. The unconfined compressive strength of cement stabilized coal gangue at each age is positively correlated with the cement dosage. The more cement dosage in the mixture, the higher the unconfined compressive strength is.

4.1.3. Indirect tensile strength

The indirect tensile strength of cement stabilized coal gangue mixture is shown in figure 10.

From figure 10, it can be seen that the indirect tensile strength of cement stabilized coal gangue mixture after 90 days shows a quadratic polynomial curve relationship with cement dosage, and it is generally positively correlated. The indirect tensile strength of cement stabilized coal gangue mixture in 90 days increases linearly with the increase of cement dosage, and slowly increases after 5% cement dosage. Tensile strength of cement stabilized coal gangue mixture is mainly composed of bonding action of cement hydration products. When the cement dosage is low, the increment of cement increases the tensile strength of the mixture more, but when the cement dosage continues to increase, the cement hydration products are sufficient to fill the gap between the
coarse and fine aggregates of coal gangue, and the tensile strength growth slows down after reaching the peak growth rate.

4.1.4. Flexural tensile strength

The relationship curve between cement dosage and the difference between 90-day flexural tensile strength and 180-day flexural tensile strength of cement stabilized coal gangue mixture is shown as figure 11.
Figure 11 shows that there is a positive correlation between cement dosage and the difference between 90-day flexural tensile strength and 180-day flexural tensile strength of cement stabilized coal gangue mixture. Late strength of cement stabilized coal gangue mixture mainly comes from hydration reaction of dicalcium silicate in cement. With the increase of cement dosage, the content of dicalcium silicate in the mixture increases, so the later strength of the mixture with high cement dosage increases more.

4.2. Durability analysis

4.2.1. Freeze-thaw performance

The frost resistance indexes of cement stabilized coal gangue mixture are BDR (compressive strength loss of specimens after ten freeze-thaw cycles) and Wn (mass change rate of specimens after ten freeze-thaw cycles). The compressive test results of each cement dosage after 180 days maintenance and 10 freeze-thaw cycles are shown in figure 12 and table 6.

It can be seen from figure 12 that with the increase of cement dosage, the mass loss caused by freeze-thaw cycle is smaller. Among them, the mass loss of cement stabilized coal gangue mixture with 3% cement dosage was 3.79%, and the mass loss decreased by 1.16% after the increase of 1% cement dosage. When the cement dosage increased to 5%, the mass loss decreased by 0.87% compared with that of 4% cement dosage, and the mass loss decreased by 0.54% when the cement dosage increased to 6%. With the increase of cement dosage, the influence of the increment of cement dosage on the mass loss before and after freeze-thaw gradually decreased.

As can be seen from table 6, the freezing resistance of cement dose to cement stabilized coal gangue mixture is generally shown as the higher the cement dose is, the higher the BDR of cement stabilized coal gangue is, so the loss of compressive strength caused by freeze-thaw cycle is lower. The strength loss of cement stabilized coal gangue with 3% cement dosage was 23.26%, and the BDR of cement stabilized coal gangue with 1% cement dosage increased by 5.31%. The strength loss of cement stabilized coal gangue with 5% cement dosage decreased by 3.64% compared with that of cement stabilized coal gangue with 4% cement dosage, and the cement dosage of cement stabilized coal gangue increased from 5% to 6%, and the BDR increased by 1.17%. With the increase of cement dosage, the increment of cement dosage has less and less effect on the increase of BDR, and the decrease of compressive strength loss in freeze-thaw cycle is less and less. The 180-day BDR of cement stabilized coal gangue mixture with 3% cement dosage meets the design requirement of «Specification for design of highway asphalt pavement» (JTG D50-2017) that the BDR in the freezing zone is greater than 70%.

Table 6. Freeze-thaw test results of cement stabilized gangue mixture.

| Cement dosage (%) | Compressive strength of unfrozen thaw cycle (MPa) | Compressive strength after freeze-thaw cycle (MPa) | BDR (%) | Compressive strength loss of freeze-thaw cycle (%) |
|-------------------|-----------------------------------------------|-----------------------------------------------|--------|-----------------------------------------------|
| 3                 | 6.42                                          | 4.92                                          | 76.64  | 23.36                                        |
| 4                 | 7.81                                          | 6.4                                           | 81.95  | 18.05                                        |
| 5                 | 9.94                                          | 8.51                                          | 85.61  | 14.39                                        |
| 6                 | 11.02                                         | 9.57                                          | 86.84  | 13.16                                        |

Figure 12. Quality loss of cement stabilized gangue mixture before and after freeze-thaw.
Figure 13. Dry shrinkage strain, single drying shrinkage coefficient, water loss rate, total shrinkage coefficient and time relationship diagram.
Table 7. Dissolution of Si$^{4+}$ and Al$^{3+}$ from spontaneous and unspontaneous combustion coal gangue.

| Specimen                              | Si$^{4+}$ dissolution (mg/g) | Al$^{3+}$ dissolution (mg/g) |
|---------------------------------------|------------------------------|------------------------------|
| spontaneous combustion coal gangue    | 2.22                         | 2.74                         |
| unspontaneous combustion coal gangue  | 0.39                         | 0.83                         |

4.2.2. Drying shrinkage

The curves of water loss, drying shrinkage strain, single drying shrinkage coefficient, general main drying shrinkage coefficient and temporal relation are shown in figures 13(a)–(d).

Figure 13(a) shows that the water loss rate of different doses of cement stabilized coal gangue mixture increases with the increase of exposure time, and the increase of water loss rate slows down and tends to be stable after 15 days. Due to the less cementitious material content in 3% cement dosage cement stabilized coal gangue mixture, the water loss is too fast, and the cement stabilized coal gangue generally shows that the higher the cement dosage, the greater the water loss rate of cement stabilized coal gangue mixture.

It can be seen from figure 13(b) that the dry shrinkage strain of coal gangue mixture stabilized by different doses of cement increases with the increase of exposure time of the specimen, and the growth of dry shrinkage strain slows down and tends to be stable after 10 days. 4%, 5%, 6% cement dosage cement stabilized coal gangue mixture showed that the higher the cement dosage, the greater the dry shrinkage strain of the mixture. 3% cement dosage cement stabilized coal gangue mixture due to the low cement dosage, the gap between aggregates can’t be completely filled by cement hydration products, and the tensile strength is low, so it will produce dry shrinkage strain higher than 4% cement dosage.

Figure 13(c) shows that there is no obvious regularity in the early stage of single drying shrinkage coefficient of each dose of cement stabilized coal gangue mixture, but with the increase of time, it starts to develop to the stable situation after 30 days, so it is not strict to only use single drying shrinkage coefficient as the evaluation index of drying shrinkage performance of cement stabilized coal gangue mixture.

It can be seen from figure 13(d) that the total shrinkage coefficient of each dose of cement stabilized coal gangue mixture generally increases first and then slowly increases, or due to the influence of the later drying shrinkage strain and the change rate of water loss rate, the total shrinkage coefficient of the mixture shows a slowly decreasing trend. The total shrinkage coefficient of 6% cement stabilized coal gangue mixture is significantly higher than that of 3%, 4% and 5% cement stabilized coal gangue mixture, showing poor drying shrinkage resistance. The total shrinkage coefficient of 4% cement stabilized coal gangue mixture is the lowest, showing the best drying shrinkage resistance.

4.3. Microcosmic and strength mechanism analysis

4.3.1. Activity analysis of coal gangue

Dissolution of Si$^{4+}$ and Al$^{3+}$ from spontaneous and unspontaneous combustion coal gangue are shown in the following table.

Table 7 shows that the amount of Si$^{4+}$ and Al$^{3+}$ ions dissolved by spontaneous combustion coal gangue is higher than that of Si$^{4+}$ and Al$^{3+}$ ions dissolved by unspontaneous combustion coal gangue, indicating that there are more free Si$^{4+}$ and Al$^{3+}$ ions in spontaneous combustion coal gangue, and the amount of active SiO$_2$ and Al$_2$O$_3$ is large. Spontaneous combustion coal gangue has better activity than unspontaneous combustion coal gangue.

4.3.2. Phase analysis of cement stabilized coal gangue mixture

The XRD patterns of cement stabilized coal gangue mixture for 7 days and 180 days are shown in figure 14.

It can be seen from figure 14(a) that in addition to the original mineral of coal gangue, the cement stabilized coal gangue mixture for 7 days and 180 days generates calcium silicate hydrate (C–S–H), gismondine (Ca$_2$Al$_4$Si$_5$O$_{18}$·4(H$_2$O)), iron silicate (Fe$_2$(SiO$_3$)$_3$), and Ca(OH)$_2$, and the cement stabilized coal gangue mixture for 7 days also contains calcium aluninate. Calcium silicate hydrate, Ca(OH)$_2$, calcium aluminate are all generated after cement hydration. Due to the instability of calcium aluninate, calcium aluminate decomposes and reorganizes over time, so there is no such mineral in 180 days cement stabilized coal gangue mixture. A new mineral phase, CaAl$_2$Si$_2$O$_6$·4(H$_2$O), was formed in the cement stabilized coal gangue mixture. Compared with the related research, it should be formed by the pozzolana reaction between the cement hydration product Ca(OH)$_2$ and the active ingredient in coal gangue. The mixture for 7 days and 180 days contained this material, indicating that the material was relatively stable and beneficial to the strength and bonding effect of the mixture. Compared with the distribution and peak strength of Ca(OH)$_2$ in 7 days and 180 days of cement stabilized coal gangue mixture, Ca(OH)$_2$ in 180 days of cement stabilized coal gangue mixture was significantly more than Ca...
(OH)_2 in 7 days of cement stabilized coal gangue mixture. The reason is that gismondine is produced by pozzolana reaction between active SiO_2, Al_2O_3 and Ca (OH)_2 in coal gangue, which consumes part of Ca (OH)_2.

4.3.3. Microstructure morphology characterization of cement stabilized coal gangue mixture

The SEM morphology of cement stabilized coal gangue mixture cured for 7 days and 180 days is shown in figures 15 and 16.

From figures 15(a), 16(a), the 100 μm interface of cement stabilized coal gangue mixture for 7 days and 180 days can be observed. The aggregate of cement stabilized coal gangue mixture for 7 days has obvious gap, and the boundary between aggregate and gelled substance is clear. Curing 180 days of cement stabilized coal gangue mixture combined closely, aggregate and gelled substance into one, can’t clearly find the boundaries of aggregate and gelled substance. This indicates that with the development of the hydration process, the hydration products and the pozzolanic reaction products are developed freely to fill the internal gap of coal gangue aggregate and the gap between coal gangue aggregate, which makes the unit structure close and has good integrity.

It can be observed from the 10 μm interface of cement stabilized coal gangue mixture for curing 7 days in figure 15(b) that the cement hydration products are mainly fibrous C–S–H gel, and fibrous C–S–H gel forms flocculent structure with other gelled substance such as ettringite, and gradually hardening into crystal structure. No Ca (OH)_2 crystal was found in the cement stabilized coal gangue mixture after 7 days of curing. Due to the

Figure 14. XRD map of cement stabilized gangue mixture at different ages.

Figure 15. SEM morphology of cement stabilized coal gangue mixture for 7 days.
pozzolanic reaction of active SiO$_2$ and Al$_2$O$_3$ in spontaneous combustion coal gangue with calcium hydroxide, calcium aluminum silicate hydrate is formed. It can be seen from figure 16 (b) that the dense structure of fibrous C–S–H gel and other gelled substance such as acicular crystal ettringite (AFT) can be observed at the 10 $\mu$m interface of cement stabilized coal gangue mixture for 180 days of curing, and it is closely bonded with coal gangue aggregate. Since the number of active SiO$_2$ and Al$_2$O$_3$ of spontaneous combustion coal gangue in cement stabilized coal gangue mixture is limited, and the hydration reaction of cement has been carried out continuously, the number of active SiO$_2$ and Al$_2$O$_3$ is insufficient in the late stage, and the generated Ca(OH)$_2$ forms crystals, which are attached to the surface of coal gangue and hydration products in a lamellar structure.

4.3.4. Mechanism of strength of cement stabilized coal gangue

According to the unconfined compressive strength data of cement stabilized coal gangue mixture from 7 to 180 days, it is found that the strength growth rate of cement stabilized coal gangue mixture from 7 to 28 days is higher, and the strength growth rate from 28 to 180 days is slow. Different cement dosage cement stabilized coal gangue mixture 7 days strength as figure 17, different cement dosage cement stabilized coal gangue mixture 7–28 days strength growth as figure 18.

From figure 17, it can be found that the unconfined compressive strength of 3% cement stabilized coal gangue mixture for 7 days is 3.26 MPa, and that of 4% cement stabilized coal gangue mixture for 7 days is 4.16 MPa, which is 27.6% higher than that of 3% cement dosage mixture. The unconfined compressive strength of 5% cement stabilized coal gangue mixture for 7 days is 5.02 MPa, which is 20.7% higher than that of 4% cement dosage mixture. The unconfined compressive strength of 6% cement stabilized coal gangue mixture for 7 days is 5.51 MPa, which is 9.7% higher than that of 4% cement dosage mixture. When the cement dosage is 4%, the strength growth rate is the highest, because in the 3% cement stabilized coal gangue mixture, due to the low
cement dosage and less gum materials, the good combination between aggregates can’t be achieved. When the cement dosage was increased by 1%, the gum materials generated can make the combination of each unit of the mixture more closely, so the strength was improved. When the cement dosage increases from 4% to 5%, the effective gum material of closely bonded aggregate increases less, so the strength increase rate decreases.

It can be seen from figure 18 that the strength growth rates of 3%, 4%, 5% and 6% cement stabilized coal gangue mixture in 7–14 days are 28.83%, 36.06%, 30.28% and 32.49%, respectively. Since the amount of Ca(OH)$_2$ generated by 4% cement dosage is sufficient to tack place pozzolana reaction with active SiO$_2$ and Al$_2$O$_3$ in coal gangue aggregate, the strength increase of 4% cement dosage in 7–14 days is the highest.

Through SEM observation of 3% cement dosage cement stabilized coal gangue mixture and 5% cement dosage cement stabilized coal gangue mixture, such as figure 19.

From figure 19, it can be clearly observed that 3% cement dosage cement stabilized coal gangue mixture has obvious pozzolana reaction, no cubic flake Ca(OH)$_2$ was found, and 5% cement dosage cement stabilized coal gangue mixture has obvious pozzolana reaction and cubic flake Ca(OH)$_2$, indicating that Ca(OH)$_2$ generated by 5% cement hydration reaction is enough to react with active SiO$_2$ and Al$_2$O$_3$ in coal gangue aggregate, cubic flake Ca(OH)$_2$ still has surplus.

In summary, from the compressive strength of cement stabilized coal gangue and pozzolanic point of view of active substances in cement stabilized coal gangue and cement hydration products, the composition of coal gangue aggregate is that coal gangue aggregate is greater than the particle size of 2.36 mm using unspontaneous combustion coal gangue aggregate, less than the particle size of 2.36 mm using unspontaneous combustion coal.
gangue aggregate: spontaneous combustion coal gangue aggregate = 1:2 (BNS:SNS:SSC = 71.26:9.41:18.83), 4% cement dosage is the most efficient for its strength growth [15, 25–27].

5. Conclusion

(1) Compared with unspontaneous combustion coal gangue, spontaneous combustion coal gangue has higher activity. The microstructure of unspontaneous combustion coal gangue is closely, and the microstructure of spontaneous combustion coal gangue has more voids. After spontaneous combustion and heavy crystallization, the active feldspar compositions of coal gangue increase. After crushing, the coarse aggregate of unspontaneous combustion coal gangue has good aggregate characteristics, which meets the requirements of pavement base for aggregate.

(2) When coal gangue aggregate composition is BNS:SNS:SSC = 71.26:9.41:18.83, cement stabilized coal gangue mixture shows optimal compressive strength. Cement stabilized coal gangue mixture with 4% cement content has the highest compressive strength growth rate from 7 days to 14 days. With the increase of time, the cement hydration reaction slows down, the volcanic ash reaction stops, and the compressive strength of the mixture increases slowly. 4% cement dosage is the best cement dosage for cement stabilized coal gangue mixture.

(3) The more the cement dosage, the higher the frost resistance index BDR of cement stabilized coal gangue. When the cement dosage is greater than 4%, the higher the cement content, the greater the dry shrinkage strain of the mixture. When the cement content is 3%, the dry shrinkage strain of the mixture is higher than that of 4% cement dosage. The total dry shrinkage coefficient of 4% cement dosage cement stabilized coal gangue mixture is the lowest, reflecting the best dry shrinkage resistance.

(4) With the increase of time, the degree of hydration gradually deepened, the better the overall combination effect of the mixture. The number of gelled substance on the aggregate surface of the 180-day cement stabilized coal gangue mixture is significantly higher than that of the 7-day cement stabilized coal gangue mixture. With the development of time, the degree of hydration is gradually deepened, and the binding of gelled substance and aggregates is closer. There is no obvious gap between aggregates, and the integrity of the mixture is enhanced, which can show better road performance.

(5) Calcium silicate hydrate (C–S–H), (CaAl2Si2O8·4(H2O)) and other materials are generated in addition to the original minerals of coal gangue in cement stabilized coal gangue mixture. The hydration product Ca(OH)2 of cement in cement stabilized coal gangue mixture takes place pozzolana reaction with active SiO2 and Al2O3 in coal gangue to form CaAl2Si2O8·4(H2O), which is beneficial to the overall strength and bonding effect of the mixture.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

CRediT authorship contribution statement

Z Li: Conceptualization, Project administration, Supervision, Writing-Review & Editing. T Guo: Conceptualization, Formal analysis, Methodology, Visualization. Y Chen: Supervision, Project administration, Data curation, Formal analysis. X Zhao: Conceptualization, Writing-Original Draft, Supervision, Investigation. Y Chen: Conceptualization, Supervision, Writing-Original Draft. X Yang: Conceptualization, Project administration, Supervision, Investigation. J Wang: Funding acquisition, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
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