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Experimental study on nano silica modified cement base grouting reinforcement materials

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Abstract. With the increasing number of underground projects, the problem of rock-water coupling catastrophe has increasingly become the focus of safety. Grouting reinforcement is gradually applied in subway, tunnel, bridge reinforcement, coal mine floor and other construction projects. At present, cement-based grouting materials are easy to shrink and have low strength after solidification. In order to overcome the special problems of high water pressure and high in-situ stress in deep part and improve the reinforcement effect. In view of the mining conditions of deep surrounding rock, a new type of cement-based reinforcement material was developed. We analyses the principle and main indexes of floor strengthening, and tests and optimizes the indexes and proportions of the two materials through laboratory tests. Then, observes and compares the microstructures of the optimized floor strengthening materials with those of the traditional strengthening materials through scanning electron microscopy. The test results show that 42.5 Portland cement-based grouting reinforcement material has the advantages of slight expansion, anti-dry-shrinkage, high compressive strength and high density when the water-cement ratio is 0.4, the content of bentonite is 4%, and the content of Nano Silica is 2.5%. The reinforcement effect is better than other traditional grouting reinforcement materials.

Keywords: floor reinforcement with grunting; nano silica; scanning electron microscope

1. Introduction

China is a country with large resources and population, and its urbanization is developing rapidly. With the exploitation of resources and the increase of subway tunnels, we are also facing various rock-water coupling problems (Sun *et al.* 2019, Zhao *et al.* 2020). With the continuous exploitation of coal resources, shallow and central coal resources are becoming less and less, and coal production is becoming increasingly tense. We have to dig deep, which facing the problems of high water pressure, high stress, etc. At the same time, China is one of the countries with the most serious mine water hazards in the world. According to the statistics of the State Administration of Safety Management, water hazards are the major disasters of coal mines which are second only to gas explosion (Wu 2014, Zhang *et al.* 2019). On the one hand, with the increase of mining depth and surrounding rock stress, joints and cracks in the fractured coal and rock mass develop, forming underground watercourse (Cheng *et al.* 2019, Kong *et al.* 2019, Sun *et al.* 2019). Influenced by the confined water of the floor, the mine faces the danger of floor water invasion. There are two main ways to solve the water inrush from the floor, one is water drainage and depressurization, the other is grouting to transform the floor serious water-bearing layer into a weak water-bearing layer or an aquifuge (Xu *et al.* 2014a, Song 2008). On the other hand, since entering the new century, urban rail transit has developed rapidly. The main structure of Metro and its subsidiary structure are designed with shallow buried depth and poor stratum conditions, which bring great difficulties to Metro construction. Grouting reinforcement of metro tunnels is one of the effective methods to ensure the stability and safety of tunnel excavation (Cheng *et al.* 2019, Zhang *et al.* 2018a, Zhang *et al.* 2018b).

The selection of grouting material is the key factor to determine the quality of reinforcement. The design of grouting material becomes particularly important. It is necessary to consider not only the cost but also the effect to reinforce the coal seam floor or tunnel (Samaila *et al.* 2019, Li 2016). Cement-based grouting materials have the characteristics of wide source of raw materials, low cost and simple operation, so cement-based grouting materials are mostly used in grouting engineering. But the cement material is easy to shrink and form cracks between the cement material and the reinforced rock mass. Therefore, a kind of cement-based grouting material with slight expansion and anti-dry-shrinkage resistance is needed. Ge (2018) summarized the change rule of grouting slurry

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performance under the action of different bentonite and admixture content through experiments. Although the problem of cement shrinkage was solved, the consolidation strength of the material was affected. Wang *et al.* studied nanocomposite grouting materials to improve the rheological properties and strength of materials. It is found that nanometer materials can improve the fluidity and strength of materials to a certain extent (Wang *et al.* 2018). Guo *et al.* developed water Glass-Nano-Silica composite polyurethane grouting material with sodium silicate and Nano Silica as main raw materials to reduce cost by adding sodium silicate to polyurethane, but reduced the properties of polyurethane materials (Guo *et al.* 2018). Taha added carbon nanotubes and carbon nanofibers to soil samples. And he find nanocarbons can improve compressive strength, Young's modulus and indirect tensile strength of soil (Taha *et al.* 2018). Morsy found that nano-scale metakaolin is highly effective in enhancing the compressive strength of mortar (Morsy *et al.*2018). It can be seen that nanometer materials can improve the strength of materials very well. Wang *et al.* studied a composite grout matrials which have the properties of easy flow and post-hardening, and he designed a visible flowing test to explore the flow characteristics of composite grout in porous media (Wang *et al.* 2019). Liu *et al.* select water-soluble polyurethane as grouting material, and add hydroxypropyl methyl cellulose to modified. It makes the anti-scour ability of the grouting material more enhanced (Liu *et al.* 2019). Seo *et al.* use aramid fiber and blast furnace slag powder to enhance the compressive strength of grout materials. Seo found that 1% increase of aramid fiber led to 1.3 times greater uniaxial compression intensity (Seo *et al.* 2019). Ni *et al.* added clay to cement-based grouting material, which reduced the cost of grouting material and improved the water resistance and plasticity of the material (Ni *et al.* 2005).

Feng (2012) found that the drying shrinkage of cement concrete with Nano Silica depends on the particle size and specific surface area of nano-SiO_2. The smaller the particle size of Nano Silica, the larger the specific surface area, the more significant the drying shrinkage of concrete. It can be seen that Nano Silica will aggravate the drying shrinkage of concrete, which is also the problem we need to solve. Zhang *et al.* have developed Polymer Foaming (PF) grouting material by themselves (Zhang *et al.* 2019). The results of scanning electron microscopy and macro-mechanical experiments show that PF grouting material is denser among molecules and its reinforcement effect is better than that of ordinary cement materials. The density between molecules is an important factor affecting the properties of materials. Zhang *et al.* studied a kind of cement-bentonite grouting material (Zhang *et al.* 2018c). The research showed that bentonite can improve the stability and erosion resistance of the material, and can effectively prevent grouting material from being washed away by groundwater. Alyousef *et al.* added marble powder as additive to self-filling concrete, and tested the properties of the material (Alyousef *et al.* 2019). The results show that marble powder improves the compactness, hardening performance and waste utilization of concrete. Grizelda *et al.* studied the influence of fly ash on cement composites, and proved that fly ash can improve the early strength of the materials (Grizelda *et al.* 2018). The effects of limestone powder on rheological properties, drying shrinkage and mechanical properties of alkali-activated slag/fly ash grouting materials (AASFGM) were studied by (Xiang *et al.* 2018). It was found that the incorporation of fine limestone powder into AASFGM has a positive effect on rheology, strength and shrinkage properties.

In addition, Groundwater can cause many hazardous problems when a tunnel is excavating. Especially, under high groundwater table condition, the seepage situation becomes more complex and it is more difficult to control the leakage of groundwater to flow into a tunnel. Grouting reinforcement can effectively control underground water flooding into the tunnel and tunnel section deformation (Packer *et al.* 2018). Gong *et al.* used the new super-fine cement grouting material and the method of multiple grouting to effectively control the surrounding rock deformation and water inflow(Gong *et al.* 2018). Yu (2013) took the No. 5 East Fourth Station of the Beijing Metro Line 6 to the project background, the combination of numerical simulation and field test proves that the pre-reinforcement of the tunnel helps to ensure the stability of the tunnel during normal construction and construction. Grouting material plays an important role in the stability of underground engineering.

In this study, ordinary Portland cement was used as the main material, and additives were selected to be added to Portland cement. We are confronted with the problems of easy shrinkage and low consolidation strength of cement base plate reinforcement materials. In order to increase the consolidation strength and compactness of the material and realize the slight expansion of the material, the material optimization was studied. Through laboratory tests, the expansibility, fluidity, curing strength and compactness of materials with different proportions are discussed, and the optimum proportions of materials are obtained.

2. Principle of floor reinforcement and main indicators for testing grouting material performance

The main cause of floor water inrush is the development of fractured zone and fissures in coal seam floor due to mining induced (Xu *et al.* 2014b). The mining action makes the rock mass under the goaf of coal seam floor in the state of free surface, and the stress state also changes, from three-dimensional stress state to two-dimensional stress or one-way stress state. When the stress above the floor is relieved, the coal seam floor will be deformed and destroyed, and the water inrush channel will be formed by the development of fracture zones and fissures. If there is a confined aquifer in the floor, it will threaten the mine production. Coal mining results in the change of stress state of surrounding rock, and the failure of rock mass produces cracks. Especially for deep seams with high water pressure and high stress condition of surrounding rock, confined water seeps and expands in cracks, reaching certain conditions, which will cause dynamic disasters such as water inrush from floor rock mass of coal seam.

Mining induced effect is inevitable, because mining induced stress will occur as long as we carry out underground excavation. Grouting reinforcement of floor is

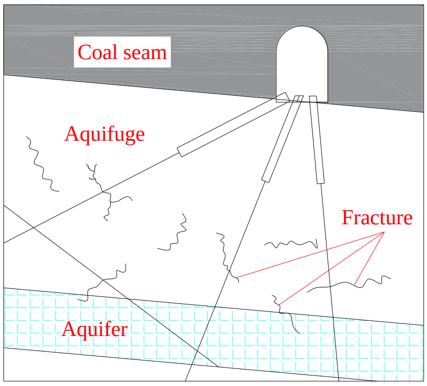


Fig. 1 Schematic diagram of grouting reinforcement

a good choice to prevent water inrush from floor (Zhang *et al.* 2018d). Grouting reinforcement is an effective measure to solve water inrush from floor in abnormal area during mining and tunnel excavation. The principle is to construct a certain amount of grouting boreholes in the lower or upper strata of working strata according to geological conditions, and then inject the mixed grouting fluid into the strata to be reinforced to fill the cracks in the strata. In this way, the grout can be used to replace part of the water in the aquifer to reduce the water-rich of the confined aquifer. At the same time, the condensated grout can quickly fix the aquifer faults and the water-proof roof together, so that the floor can be strengthened, and the original confined aquifer can be turned into a weak aquifer, thus reducing the risk of water inrush from the floor. The grouting schematic diagram is shown in Fig. 1. Grouting material is an important factor affecting the effect of grouting reinforcement, so it is very important to design a good grouting material.

The main properties and testing methods of grouting materials are as follows:

(1) Expansion property: Measure macro-expansion of material by beaker.

(2) Fluidity: Measuring fluidity with standard truncated cone circular die.

(3) Compressive strength: The laboratory 119 servo press was used to test the compressive strength of the stone body with the size of 70.7mm*70.7mm*70.7mm. The test method refers to "Method for Testing the Strength of Cement Mortar (ISO Method)" (GB/T 17671-1999).

(4) Slurry relative density: JSM-6510LV high and low vacuum scanning electron microscope was used. Microscopic comparison was carried out by scanning electron microscopy to observe the structure and cracks of sealing materials in the reaction process.

3. Experiments, results and discussions

3.1 Material selection

Because of the large amount of floor reinforcement, it is necessary to consider the raw material and cost. Therefore, ordinary 42.5 Portland cement, which is easy to obtain and relatively inexpensive in price, is chosen as the main material. Fluidity is one of the main indicators of material performance. Calcium hydroxide colloidal structure formed by lime particles in lime slurry after lime maturation can absorb a large amount of water, so we mix a small amount of quicklime into the material to improve its workability (It includes three aspects: fluidity, water retention and cohesion.). As a floor reinforcement material, the strength of the material is particularly important, which is also the focus of this study. The strength of the material is closely related to the density and compressive strength of the material. Nano Silica (SP30T) can reduce the number of hardened cement pastes and the porosity in the transition zone, thereby improving the density, aging resistance, strength and chemical resistance of cement materials. So we choose Nano Silica as one of the excipients of materials. Expansion performance, fluidity and compressive strength of materials were tested in laboratory to determine the appropriate material ratio, and then examined by scanning electron microscopy. Finally, the grouting material that meets the requirements is obtained. The main components of the new sealing material are shown in Fig. 2 above.

Fig. 2 Main components of grouting reinforcement materials

Table 1 Material ratio

Level	Bentonite (%)	Nano Silica (%)
1	0	0
2	2	1
3	4	2
4	6	3
5	8	4

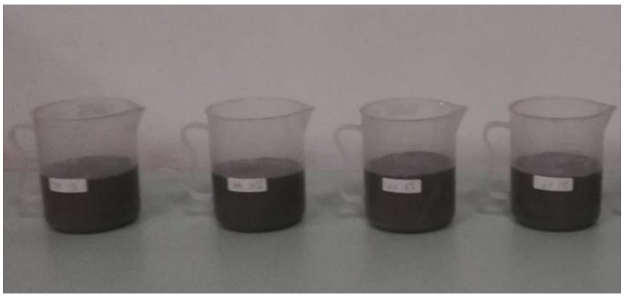


Fig. 3 Test for expansion performance

3.2 Experimental scheme

Water cement ratio is an important factor affecting the performance of materials, so we need to determine the

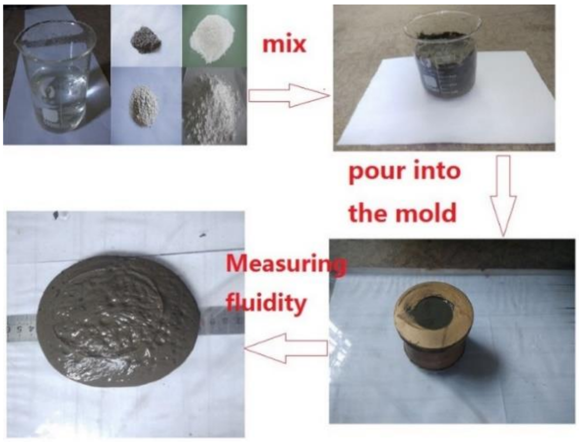


Fig. 4 Flow chart for fluidity measurement

water cement ratio of materials. We configure four kinds of cement slurries with water-cement ratio of 0.3, 0.4, 0.5 and 0.6 respectively, and choose the best water-cement ratio. Then the orthogonal test of the remaining factors is carried out on the basis of determining the optimal water cement ratio. The main factors affecting the properties of the materials are the amount of bentonite and Nano Silica. Five levels of 25 groups are selected for each factor, and the amount of each factor is shown in Table 1.

3.2.1 Expansion experiment

Expansion performance test uses 500 ml beaker. 300 ml slurry is poured into the beaker. Four specimens are made according to the ratio in Table 1, as shown in Fig.3. Maintenance in a constant temperature box at 25-30 C. The volume values are read every 5 minutes and recorded as V_1, V_2, V_3, V_4 …V_n respectively, and V_n is the final volume value. The final volume of the test record is taken as the average of four specimens.

3.2.2 Fluidity test

Steps: Place the glass plate on the horizontal position, rub the glass plate, the truncated circular die, the mixer and the mixing pot with wet cloth to make the surface wet without water. Place the truncated circular die in the center of the glass plate and cover it with wet cloth for standby use.

The stirred paste is quickly poured into the circular die of the truncated cone, and scraped flat with a scraper. The circular die of the truncated cone is lifted in the vertical direction. At the same time, the meter is opened and timed to allow the cement paste to flow on the glass plate to 30S. The maximum diameter of the two directions of the flow part is measured by a ruler, and the average value is taken as the fluidity of the cement paste. The fluidity test process is shown in Fig. 4.

3.2.3 Uniaxial compressive strength test

A layer of release agent was coated on the 70.7 mm x 70.7 mm x 70.7 mm mould. After mixing, the slurry was poured into the mould. The slurry was vibrated slightly to make it dense. It was maintained in a curing box with relative humidity of 95% and temperature of 20°C. The specimen was maintained in the mould until its predetermined age and then demoulded. The uniaxial compressive strength of the specimens maintained for 3 days and 28 days was tested by electro-hydraulic servo pressure tester. The loading speed was 0.3 MPa/s. The test was stopped after the specimens were broken. The test flow is shown in Fig. 5. According to GB177-85 “Cement mortar strength test method”, the compressive strength of composite cementing material samples was determined.

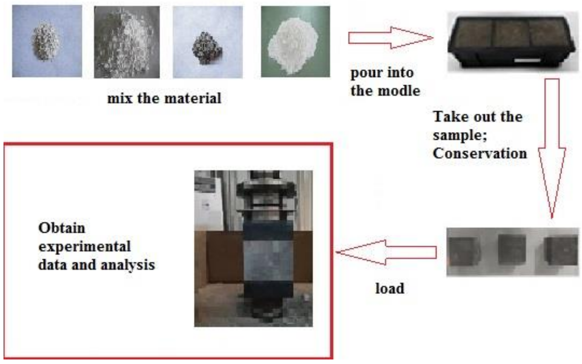


Fig. 5 Compressive strength test flow

3.3 Result and analysis

3.3.1 Expansion test

During the test, it was found that the slurry with 0.3 water cement ratio was too thick and fluidity was too poor to pump. Because the water cement ratio is too large, the slurry with 0.5 water cement ratio bleeds. A large amount of free water secretes the slurry surface, which affects the solidification and hardening of the material and decreases the water holding capacity of the material. Therefore, the water cement ratio of 0.4 is selected for the experiment. The expansion performance of the new grouting reinforcement material varies with the amount of admixture as shown in Fig. 6.

From Fig. 6 (bentonite content in the icon), it can be seen that the final volume of the material increases with the increase of bentonite content when the water cement ratio is constant. When the content of bentonite is constant, the final volume of the material decreases with the increase of Nano Silica. However, it can be seen that the effect of Nano Silica on the volume of bentonite is slight. In other words,

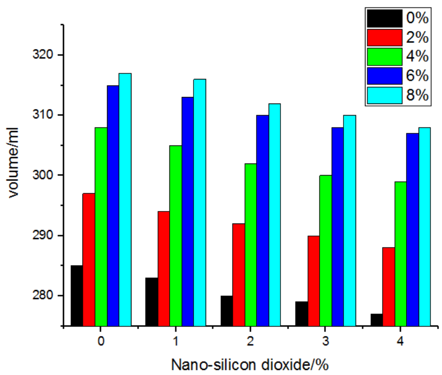


Fig. 6 Volume of material varies with composition ratio

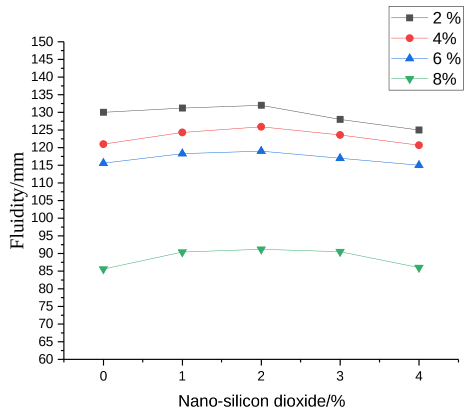


Fig. 7 Trend chart influencing slurry flow degree

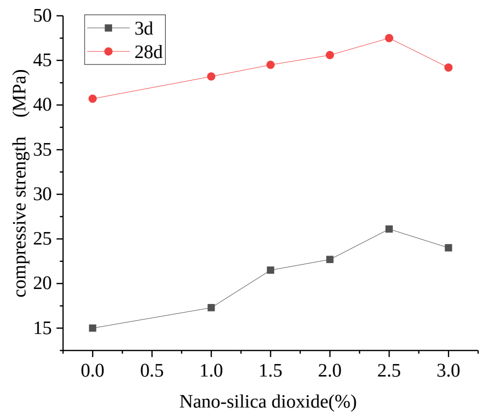


Fig. 8 Compressive strength versus Nano Silica content

the influence of Bentonite on the volume of materials is greater than that of Nano Silica. When the content of bentonite is more than 6%, the expansion rate decreases gradually. The main reason is that when the content of bentonite increases, the water cement ratio remains unchanged, the amount of free water is constant, and the water absorption of bentonite is gradually insufficient, resulting in the decrease of material expansion rate. At the same time, it can be seen that when the content of bentonite is more than 4%, the material is more than 300 ml to achieve slight expansion. Therefore, bentonite selection of 4-8% is eligible. In order to further optimize the material ratio, the fluidity test results are analyzed.

3.3.2 Fluidity

The fluidity of slurry determines the water dispersibility and diffusion resistance of slurry in grouting environment, and also affects the pumpability and injectability of slurry to a certain extent. By adjusting the size of slurry fluidity, the diffusion range of slurry can be effectively controlled, and the reinforcement area can be controlled within the required range. It is of great significance for saving engineering cost and improving construction quality. The trend diagram of slurry flow is obtained as shown in Fig. 7.

Expansion test shows that with the increase of bentonite, the plasticity of the material increases and the corresponding fluidity decreases. When the content of bentonite is more than 6%, the fluidity decreases sharply. The main reason is that the bentonite has the characteristics

Table 2 Controlled experimental test results

	Volume (ml)	Fluidity (mm)	Compressive strength 3d (MPa)	Compressive strength 28d (MPa)
Control group	285	150	19	43
Experimental group	301	125	26	47.5

of water absorption and expansion, which reduces the free water. On the other hand, the particle size of bentonite is smaller, and the water demand increases with the increase of the content of bentonite. It is found that when the water cement ratio is 0.4, the fluidity of the material can be increased when the content of Nano Silica is between 0 and 4%. The fluidity increases first and then decreases, and reaches the maximum when the content of Nano Silica is 2%. Although more than 2% Nano Silica can still increase the fluidity, some Nano Silica can not be used as filler to fill the cement particles, but absorb free water. On the other hand, it can be seen that Nano Silica has less influence on the fluidity of materials, and bentonite has greater influence on the fluidity of materials.

Expansion test and fluidity test show that when the content of bentonite is 4% and Nano Silica is 1-3%, it has better expansion and flow characteristics. In order to continue to optimize the material ratio, the compressive strength test results are analyzed.

3.3.3 Uniaxial compressive strength

Above experiments, we determined that the content of bentonite is 4%, the content of Nano Silica is 1-3%, we take Nano Silica as a single variable, the content is 0%, 1%, 1.5%, 2%, 2.5%, 3%, respectively. The curve of the compressive strength of the material with the content of Nano Silica is obtained by experiment, as shown in Fig. 8.

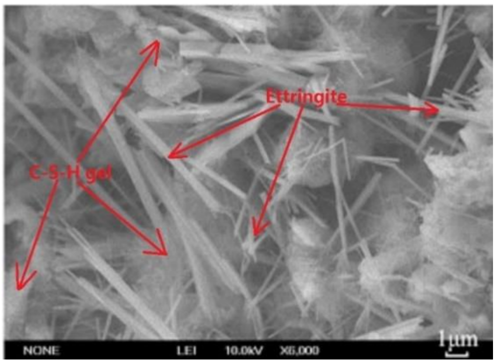
From Figure 8, it can be seen that the compressive strength of the specimens increases when the dosage is less than 2.5%, especially when the early strength (3d) increases rapidly. When the dosage is more than 2.5%, the compressive strength of the specimens decreases obviously. The results show that 2.5% Nano Silica is an appropriate addition, and Nano Silica plays an important role in improving the compressive strength of materials.

In order to study the effect of Nano silica on grouting materials, a control experiment was set up. The control group used Portland cement single clinker, and the experiment was configured according to the ratio obtained from the above test. The obtained test results are shown in Table 2.

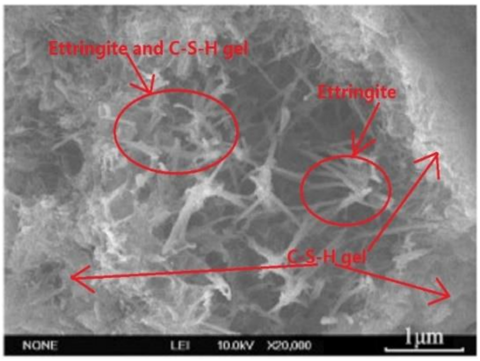
The results showed that the volume increase was 5.6%, the fluidity decreased by 16.6%, the early compressive strength (3d) increased by 36%, and the later compressive strength (28d) increased by 10.4%. It can be seen that the new grouting material has better resistance to shrinkage than ordinary grouting materials, and the compressive strength has also been improved, especially the early compressive strength has been greatly improved.

4. Microstructure comparison

When 2.5% Nano Silica is added into cement mortar,



(a) No Nano Silica added



(b) Addition of 2.5% Nano Silica

Fig. 9 Scanning electron microscopy

the age of 3D cement mortar can be observed by SEM in Fig. 9. The results show that:

(1) After adding 2.5% Nano Silica, the 3D morphology and structure of cement slurry become more compact.

(2) Needle and column ettringite are not well developed in the hydration products of 3D cement mortar reference samples, and the overlap between them is not close enough and shows loose distribution. There is no dense group with C-S-H (CxSHx-0.5) gel, a small number of pores, and a certain amount of flaky Ca $(OH)_2$ sporadic.

(3) With the addition of 2.5% Nano Silica, the columnar ettringite and C-S-H (CxSHx-0.5) become compact to some extent. C-S-H (CxSHx-0.5) gel formed clusters of clusters. Ettringite overlapped with C-S-H (CxSHx-0.5) gel.

(4) Ettringite is tightly interwoven with C-S-H (CxSHx-0.5) gel, the capillary pore size is smaller, the pore is regular, and the surface tends to be smooth. The cloud shaped C-S-H gel gathered by particles intertwined and overlapped each other, and a structure similar to the stone texture appeared. Because Nano Silica has good dispersibility, the total void fraction decreases, and the structure of cement paste is more compact. The compressive strength of the sample is obviously higher than that of the reference sample of the same age.

5. Conclusions

Focusing on the application of composite material technology to solve the problem of floor reinforcement. The grouting reinforcement materials are studied from three aspects: theoretical analysis, laboratory material development and performance test, and comparison with traditional reinforcement materials. The following conclusions are drawn:

• The main purpose of grouting reinforcement is to reinforce the roof and floor. On the one hand, it is necessary to fill the cracks, on the other hand, it is necessary to block the confined water of the floor. Therefore, the effect of the floor reinforcement largely depends on the expansion, fluidity, compressive strength and compactness of the material.

• In view of the shrinkage of cement materials after solidification and the formation of cracks between the cement materials and the reinforced rock mass, which affect the reinforcement effect, the slight expansion grouting reinforcement material is developed.

• Using 425 Portland cement as main material, bentonite and Nano Silica as additives, a new reinforcement material was made up. Through experiments, it was determined that when the water-cement ratio was 0.4, the mass content of bentonite was 4%, and the content of Nano Silica was 2.5%, the swelling property and fluidity of the material were the best, and the compressive strength of the material at early and late stages were greatly improved. It is also found that bentonite plays an important role in improving the plasticity of materials.

• Microstructure of cement mortar strengthened with 2.5% Nano Silica was observed and analyzed by SEM scanning. It can be seen that the morphology and structure of cement mortar became more compact after adding 2.5% Nano Silica. The good dispersibility of Nano Silica reduced the total voidage, the structure of cement stone was more compact, and the compressive strength was obviously higher than that of reference samples of the same age.

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Conflicts of interest

The authors declare no conflict of interest.

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