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Research

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Proportion optimization of grouting materials for roadways with soft surrounding mass

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Abstract: Severe deformation and failure frequently occur in roadways with soft or weak surrounding rock and have greatly influenced safe and efficient mining of coal in many coal mines. Using portland cement, emery and fly ash as main raw materials, through laboratory tests, effect of water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent, fly ash/cement ratio and various performance indexes of grout of fluidity, viscosity, setting time, bleeding rate, compressive strength, concretion rate and various performance indexes were systematically analyzed. An optimized mixture ratio of the main raw materials added in the grouting material proportion was determined through uniform design method, an optimal mixture ratio was determined by regression analysis. The results show that: 1) The flow performance is significantly affected by change of sodium silicate and water reducer, the compressive strength of grouting material increases significantly with increase in emery content, and decreases significantly with increase in water reducer. 2) An optimized mixture ratio among water cement ratio, cement sand ratio, water/sodium silicate ratio, water reducing agent, fly ash/cement ratio in the grouting material is 0.75, 1.2, 8%, 3% and 0.18, respectively. Field test demonstrated that the material has better performance in reinforcing weak and broken rock mass.

Key words: Laboratory grouting test; uniform design; regression analysis; mixture proportion

1 Introduction

China's coal resources are widely spread with various geological conditions. A considerable number of the roadways are driven in soft rock. Weathering, spalling, and swelling and weakening of surrounding rock lead to obvious characteristics of soft rock even in the low pre-mining stress environment (Wang et al 2012; Liu et al 2011; Feng et al 2018). And the potential risks of soft rock roadway mainly include large amount of total deformation, high convergence rate, long continuous time and

invalid anchorage. These may cause unserviceability of the roadway and other unprecedented technical problems associated with soft surrounding rock. Grouting, lining with tapered stones and shotcrete are commonly used to support soft rock roadway at home and abroad, among which grouting is the most common.

Surrounding rock grouting is one of the effective methods for roadway reinforcement support (Kang et al 2013; Meng et al 2015), especially for soft surrounding coal and rock. The grout makes weak coal and rock mass more integrated,

forming a new bearing structure, thus improving the overall stability of the weak coal and rock mass. After grouting, the cohesive on and internal friction angle of surrounding rock is increased significantly. As an important part of grouting technology, materials directly affect the final grouting result. At present, common grouting materials mainly include ordinary cement, cement with sodium silicate, cement with clay and chemical materials, among which the chemical materials are represented by the polymer materials such as polyurethane and epoxy resin (Hou et al 2013; Wang et al 2010; Han et al 2013; Cao et al 2015).

Based on characteristics of grouting projects, extensive research into new grouting materials was also carried out. Sun et al (2017) developed a grouting material that can effectively improve the quality of expansive soft surrounding rock through the test and application of anti-expansion grouting material. Shimada et al (2014) studied the effect of cement grouting material with different water-cement ratio on reinforcement result of floor using ordinary cement and a variety of additives. Liu et al (2012) made a high-performance grouting material with expansion, impermeability, moisture-proof, strengthened and ability of dynamic pressure absorption. On the basis of traditional cement materials, Zhao et al (2017) added fly ash, dust and other power plant waste residue, and found that a certain amount of minerals can improve the compressive strength of grouting material; In the study of sodium silicate grouting materials, Wu et al (2016) found that sodium silicate composite grout could significantly improve setting time and strength of grout. Feng (2010) and Xu (2014) confirmed that

polyurethane grouting materials can improve the compressive and flexural strength of composite rock mass. Gao et al (2018) prepared a new polymer grouting material with fast polymerization reaction, low viscosity, high adhesive strength and good fire-proof property. Gao et al (2015) developed a new type of polyurethane water-blocking material, which has good reinforcement properties for water-bearing fractured coal and rock mass. Li et al (2015) studied the spread mechanism of self-expanding polymer grouting material in rock mass fractures.

Above research results show that, the inorganic cement grouting material is of low cost and easy to get, but its penetrability and adhesiveness are poor which causes the existing cement material is difficult to meet the demand of engineering. Due to high cost and poor durability, chemical materials are not used extensively. To realize the grouting material performance to meet the needs of the project, being green and environmentally friendly, use available materials locally, and reducing the cost become the focus of the study. And for the grouting treatment of weak and fractured rock mass, the bearing capacity is low, so the selected material must be able to effectively strengthen weak coal and rock mass. This paper chooses the most suitable grouting material for a coal mine in Shanxi Province under its geological conditions, so as to achieve the ideal technical and economic indexes. According to the proposed technical indexes, seek the optimal mixture ratio design, to obtain an ideal grouting material which meets the requirements of grouting reinforcement in soft rock roadway.

2 Methodology and materials

2.1 Testing methods

This experiment adopts the method of uniform design, initiated by Chinese statistician professor Fang Kaitai and academician Wang Yuan of the Chinese academy of sciences (Wang et al 1996). It is the preferred method to deal with multi-factor and multi-level test design, which can complete the research and development of complex problems and new products with less test times. The biggest characteristic of uniform design is that the maximum horizontal number can be equal to the number of experiments, instead of the relationship between the square number of experimental factors, and the number of experiments is only related to the number to be investigated. However, generally speaking, the number of experiments should be about 3 times of the number of experimental factors, which is conducive to optimization and analysis.

The grout ratio test should be conducted in accordance with the relevant provisions of the test method for water consumption for standard consistency, setting time and stability of cement (GB /T 1346-2011) and the test method for strength of cement cementing sand (ISO) (GB /T 17671-2019). The mortar proportion test should be conducted in accordance with the basic performance test method of building mortar (JGJ70-2009); The proportion test of cement and fly ash sand and fine stone grout should be conducted in accordance with the standard for performance test of ordinary concrete mixtures (GB /T 50080-2016) and the standard for inspection and evaluation of concrete strength (GB /T 50107-2010).

2.2 Experiment materials

The cement used in this test is P.O 42.5r ordinary portland cement produced by

Shanxi cement factory, whose quality meets the national inspection standards; The fly ash is produced by Shanxi Yangcheng power plant. Sodium silicate with a modulus of 2.5 is used for sodium silicate; Emery is made of quartz sand with a specific gravity of 3.22 and a microhardness of 3120 kg/mm. The water-reducing agent uses the HS-HF high-efficiency water-reducing agent produced by the Second Institute of chemical industry.

2.3 Experiment indicators, factors and levels

The contributing factors (Shimada et al 2014; Liu et al 2019) of single liquid grouting considered in this paper are: water/binder ratio (Asbridge et al 2002; Yoon et al 2009), cement/sand ratio (Yoon et al 2009), water/sodium silicate ratio (Aliabdo et al 2016), water reducing agent (Zhang et al 2007) and fly ash/cement ratio (Zeng et al 2012). To analyze the performance indexes of grout and obtain the optimal mixture ratio scheme, if the test at level 5 is set for all the influencing factors, the orthogonal test design (Chen et al 2009) needs to carry out at least 25 tests, with a large number of tests. Therefore, the uniform design (Li et al 2009) table is adopted in this study, which can effectively avoid a large number of tests.

According to engineering experience and statistics, the cement-sand ratio is generally controlled between 0.9 and 1.3, the ratio between water and cementing materials is generally controlled between 0.6 and 0.8, the ratio between sodium silicate and water content is between 0 and 1/5, fly ash is about 10% to 20% of cement content, and the water-reducing agent is between 0 and 3.5% of the sum of cement and fly ash. Sodium silicate engineering

experience: in grouting engineering, the value of sodium silicate is generally controlled between 0 and 1/5 of the water content. Therefore, to simplify the test scheme, a uniform test was designed for five

factors, namely the water-binder ratio, cement-sand ratio, water /sodium silicate ratio, water reducing agent and fly ash/cement ratio. The experimental design is as follows:

Table 1 Single-liquid cement grout factor level table

Fa ctors Levels	Water-binder ratio	Cement-sand ratio	Water- sodium silicate ratio	Water reducing agent	Fly ash/cement ratio
1	0.6	0.9	0	1.5%	0.1
2	0.65	1.0	0.05	2%	0.12
3	0.7	1.1	0.1	2.5%	0.15
4	0.75	1.2	0.15	3%	0.18
5	0.8	1.3	0.2	3.5%	0.2

Table 2 $U_{15}(15^5)$ Uniform design table

Batch	1	2	3	4	5
1	1	1	4	2	3
2	2	3	2	2	5
3	3	5	3	5	2
4	4	2	1	3	2
5	5	3	5	1	1
6	1	4	4	4	4
7	2	1	2	4	5
8	3	2	5	5	3
9	4	5	1	1	3
10	5	2	3	1	4
11	1	4	2	4	1
12	2	5	5	3	5
13	3	1	3	3	1
14	4	3	1	5	4
15	5	4	4	2	2

Table 3 Single liquid uniform test design table

Mixture number	Water-binder ratio(x_1)	Cement-sand ratio(x_2)	Water- sodium silicate ratio(x_3)	Water reducing agent(x_4)	Fly ash/cement ratio(x_5)
1	0.6	0.9	0.15	2%	0.15
2	0.65	1.1	0.05	2%	0.2
3	0.7	1.3	0.1	3.5%	0.12
4	0.75	1.0	0	2.5%	0.12
5	0.8	1.1	0.2	1.5%	0.1
6	0.6	1.2	0.15	3%	0.18
7	0.65	0.9	0.05	3%	0.2

8	0.7	1.0	0.2	3.5%	0.15
9	0.75	1.3	0	1.5%	0.15
10	0.8	1.0	0.1	1.5%	0.18
11	0.6	1.2	0.05	3%	0.1
12	0.65	1.3	0.2	2.5%	0.2
13	0.7	0.9	0.1	2.5%	0.1
14	0.75	1.1	0	3.5%	0.18
15	0.8	1.2	0.15	2%	0.12

2.4 Experiment process

According to Table 4 of the uniform test design ratio, different grouting materials were selected for the grouting mixture ratio test. The evaluation parameters of the mixture ratio test include: slurry fluidity (Khayat et al 1998), viscosity(Kim et al 2009), initial setting time, final setting time (Khayat et al 2002), bleeding rate (Gustin et al 2007), compressive strength (Mujah et al

2016) and concretion rate (Xue et al 2013). The test mainly examines the influence of different grouting materials and different mixing ratios on the above evaluation index, and a reasonable grouting slurry mixing ratio is determined to provide a basis for the grouting treatment of soft rock roadways.

The preparation of grouting materials and research procedures are shown in Fig. 1.

Table 4 Single-liquid cement grout uniform test design proportion table

Mixture number	Cement	Fly-ash	Sodium silicate	Emery	Water	water reducing agent
1	434.8	65.2	45.0	555.6	300	10
2	416.7	83.3	16.3	454.5	325	10
3	446.4	53.6	35.0	384.6	350	17.5
4	446.4	53.6	0.0	500	375	12.5
5	454.5	45.5	80.0	454.5	400	7.5
6	423.7	76.3	45.0	416.7	300	15
7	416.7	83.3	16.3	555.6	325	15
8	434.8	65.2	70.0	500	350	17.5
9	434.8	65.2	0.0	384.6	375	7.5
10	423.7	76.3	40.0	500	400	7.5
11	454.5	45.5	15.0	416.7	300	15
12	416.7	83.3	65.0	384.6	325	12.5
13	454.5	45.5	35.0	555.6	350	12.5
14	423.7	76.3	0.0	454.5	375	17.5
15	446.4	53.6	60.0	416.7	400	10

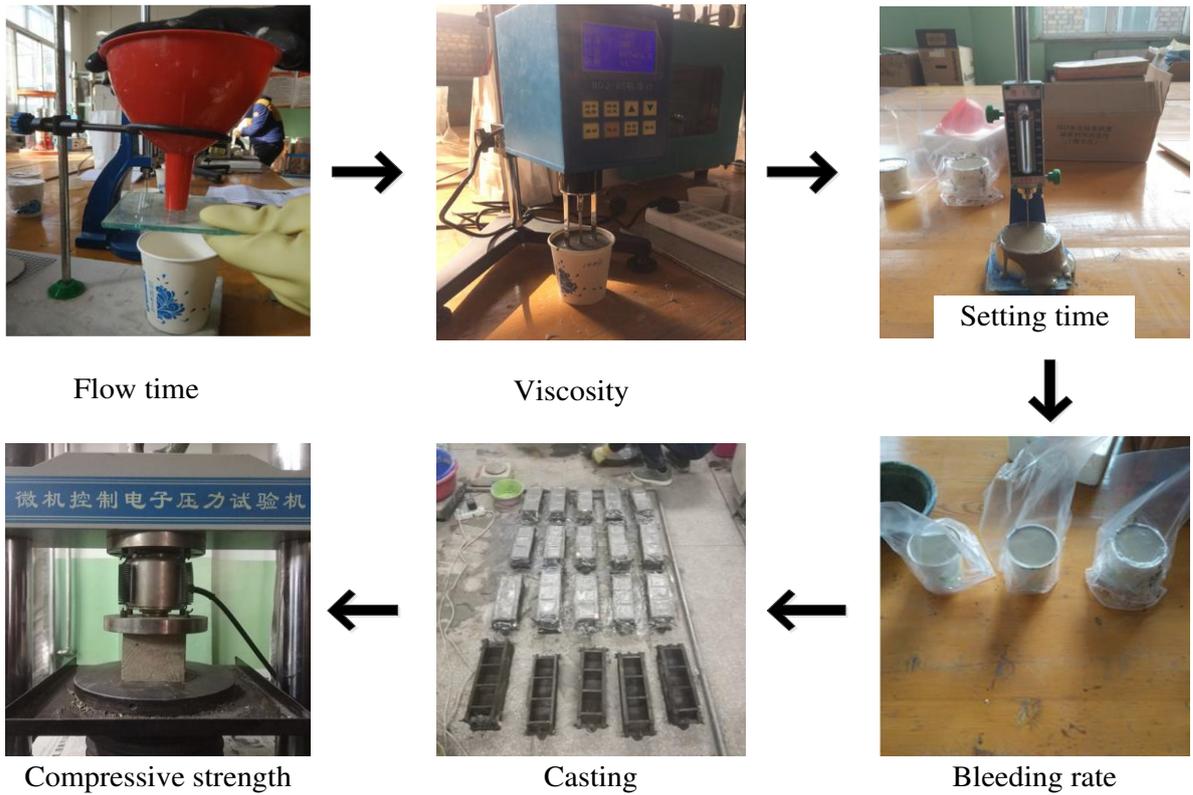


Fig. 1 Test steps

3 Results and discussion

Based on uniform design test scheme, the test results obtained are shown in table 5.

3.1 Experimental and regression analysis results

Table 5 Grouting materials uniform design test results

Test batch	Flow time (s)	Viscosity (mPa.S)	Setting time		Bleeding rate(%)	Compressive strength				Concretion rate(%)
			Initial (min)	Final (min)		1d (MPa)	3 d (MPa)	7 d (MPa)	28 d (MPa)	
1	18.9	5413	1	15	1.60	14.72	17.38	23.12	28.41	98.50
2	12.5	2785	10	40	4.70	11.54	15.05	20.23	25.14	94.82
3	14.2	3164	15	55	4.80	12.89	15.85	20.78	25.65	95.98
4	7.3	578	180	255	8.40	14.13	16.67	22.79	28.02	87.80
5	19.8	5765	0.5	10	0.00	14.53	17.64	23.38	28.34	100.00
6	15.8	4964	1	15	1.50	12.01	15.58	20.79	25.67	98.80
7	9.1	1386	20	75	5.30	12.34	15.87	21.36	26.14	93.70
8	15.7	3893	5	20	1.20	13.94	17.05	22.87	27.82	94.20
9	7.6	354	165	235	5.20	11.54	14.32	19.13	23.91	90.54

10	14.9	3548	10	35	2.70	13.82	16.94	23.05	28.17	96.60
11	10.6	2176	20	80	6.30	10.27	13.82	19.28	24.35	93.42
12	16.9	4265	2	15	0.50	14.2	17.24	22.93	27.87	99.20
13	14.5	3364	12	45	3.80	13.76	16.48	22.46	27.74	96.30
14	7.2	486	295	410	7.80	12.61	16.19	21.65	26.37	86.40
15	19.2	5589	0.5	10	0.50	13.13	16.52	22.05	26.78	99.70

According to the test results in the table, the material mixture ratio was taken as content ratio for multivariate nonlinear regression analysis (Thanoon et al 2008). The model adopted the following second-order mixing material normal polynomial (Nia et al 2017), and SPSS software (Sonebi et al 2013) was used for regression analysis:

$$y = b_0 + \sum_{k=1}^5 b_k x_k + \sum_{j=1}^5 \sum_{k=j}^5 b_{jk} x_j x_k + \varepsilon \quad (1)$$

Where x_j , x_k is the test factor, b_0 , b_k , b_{jk} is the regression coefficient, x_1 is the water/binder ratio, x_2 is the cement/sand ratio, x_3 is the water/sodium silicate ratio, x_4 is the water reducing agent content and x_5 is the fly ash/cement ratio. According to the test results, the regression relationship is as follows:

Flow empty time:

$$f_{flowability} = -207.59 + 364.73x_1 + 84.08x_2 + 191.3x_3 + 1076.78x_4 + 391.28x_5 - 68.91x_1^2 - 33.9x_2^2 - 309.75x_3^2 - 20202.79x_4^2 - 1306.17x_5^2 + 78.19x_1x_3 - 53.34x_2x_3 - 2687.98x_3x_4 \quad (2)$$

Correlation coefficient R=0.9929

The adjusted correlation coefficient Ra=0.9814

Viscosity:

$$f_{viscosity} = 5363.59 - 43058.78x_1 + 46463.39x_2 + 29832.15x_3 - 1035260x_4 + 27693.78x_5$$

$$+13397.74x_1^2 - 21567.01x_2^2 - 181201x_3^2 + 2662020x_4^2 - 251637x_5^2 + 821426x_1x_4 + 25294.85x_2x_3 + 1596440x_4x_5$$

(3)

Correlation coefficient R=0.9995

The adjusted correlation coefficient Ra=0.9878

Initial setting time:

$$f_{initial\ setting} = 3661.03 - 6675.3x_1 - 2025.98x_2 - 1493.18x_3 - 6843.16x_4 - 2353.04x_5 + 3802x_1^2 + 69x_2^2 + 97x_3^2 - 16305.59x_4^2 + 1463.76x_1x_2 + 6834.36x_2x_3 - 35590.8x_3x_4$$

(4)

Correlation coefficient R=0.9994

The adjusted correlation coefficient Ra=0.9960

Final setting time:

$$f_{final\ setting} = 7518.55 - 12953.51x_1 - 3634.62x_2 - 3028x_3 - 249x_4 - 69x_5 + 6936.78x_1^2 + 8162.05x_3^2 + 715519x_4^2 - 1285.99x_5^2 + 3289.96x_1x_2 + 8883.31x_2x_3 - 37621x_3x_4 - 60x_4^2$$

(5)

Correlation coefficient R=0.9998

The adjusted correlation coefficient Ra=0.9887

Bleeding rate:

$$f_{bleeding} = 1.79 + 3.57x_1 + 0.69x_2 - 0.14x_3 + 5.67x_4 + 2.76x_5 - 1.97x_1^2 + 0.89x_3^2 - 65.56x_4^2 - 5.32x_5^2 - 0.8x_1x_2 - 0.96x_2x_5 - 13.35x_3x_4 - 8.67x_4x_5 \quad (6)$$

Correlation coefficient R=0.9995

The adjusted correlation coefficient Ra=0.9875

1d Compressive strength:

$$f_{1d} = -33.52 + 188.82x_1 - 55.86x_2 - 3.92x_3 + 53.46x_4 + 149.83x_5 - 122.88x_1^2 + 15.58x_2^2 + 140.65x_3^2 - 10340.43x_4^2 - 490.01x_5^2 - 418.42x_1x_4 + 699.04x_2x_4 - 562.89x_3x_4 \quad (7)$$

Correlation coefficient R=0.9910

The adjusted correlation coefficient Ra=0.9842

3d Compressive strength:

$$f_{3d} = 10.28 + 62x_1 - 38.68x_2 + 0.54x_3 - 141.54x_4 + 73.56x_5 - 38.6x_1^2 + 10.62x_2^2 + 99.76x_3^2 - 223.97x_5^2 - 6918.46x_4^2 + 475.4x_2x_4 - 420.1x_3x_4 \quad (8)$$

Correlation coefficient R=0.9990

The adjusted correlation coefficient Ra=0.9931

7d Compressive strength:

$$f_{7d} = 46.56 + 12.19x_1 - 53.62x_2 - 21.17x_3 + 28.96x_5 - 273.45x_4 + 13.23x_2^2 + 158.56x_3^2 - 10876.15x_4^2 - 74.16x_5^2 + 750.88x_2x_4 \quad (9)$$

Correlation coefficient R=0.9961

The adjusted correlation coefficient Ra=0.9864

28d Compressive strength:

$$f_{28d} = 62.54 + 2.22x_1 - 59.84x_2 - 24.62x_3 - 18.56x_5 - 258.58x_4 + 14.78x_2^2 + 180.36x_3^2 - 13310.51x_4^2 - 73.8x_5^2 + 66x_1x_5 + 8x_2x_3 \quad (10)$$

Correlation coefficient R=0.9991

The adjusted correlation coefficient Ra=0.9956

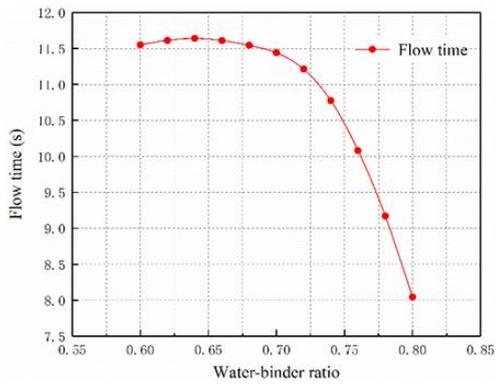
Concretion rate:

$$f_{consolidation} = 0.65 + 0.9x_1 + 0.064x_2 + 1.49x_3 - 3.48x_4 - 0.76x_5 - 0.69x_1^2 - 4.78x_3^2 - 56.33x_4^2 - 3.32x_3x_4 + 30.57x_4x_5 \quad (11)$$

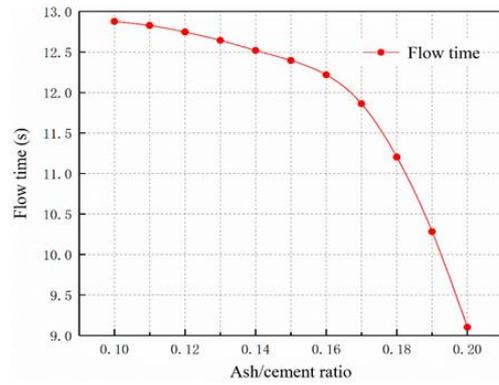
Correlation coefficient R=0.9960

The adjusted correlation coefficient Ra=0.9861

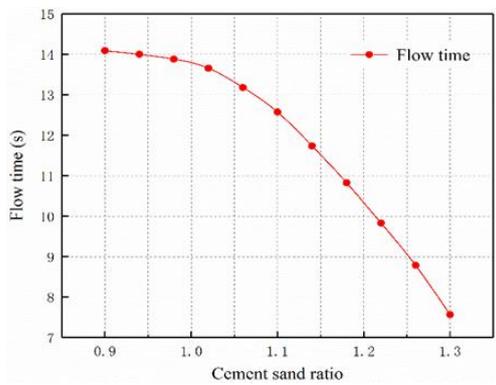
The single factor influence analysis (Sui et al 2015) was carried out for each performance index and influence factor of grouting material. Based on the mixture ratio of group 7, ($x=0.65$, $x=0.9$, $x=0.05$, $x=3\%$, $x=0.12$) change the value of one of the influencing factors, study the influence change relation of the influencing factor on fluidity, viscosity, initial and final setting time, bleeding rate, 1d, 3d, 7d, 28d compressive strength and concretion rate, and obtain the following series of change relationship curves:



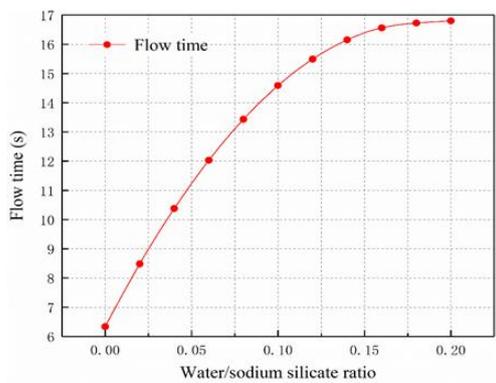
(a)



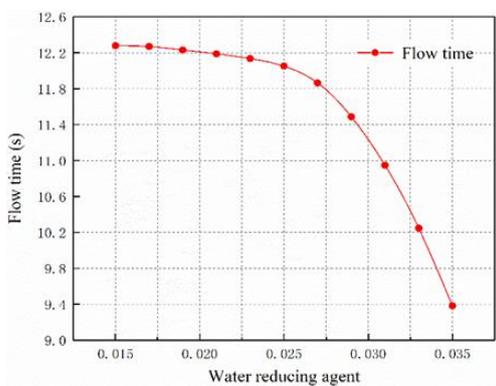
(e)



(b)



(c)



(d)

Fig. 2 The relationship between grout flow time and various influencing factors

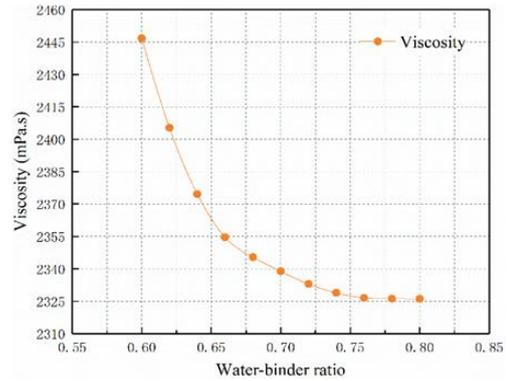
Fig. 2 shows that when the experimental range of each influence factor is given, the fluidity of the slurry is mainly related to the changes of sodium silicate, water reducing agent, water/binder ratio, cement/sand ratio, and fly ash/cement ratio. Among them, the changes in the content of sodium silicate and cement-sand ratio have a significant effect on the flow time. When the content of sodium silicate is too large, the slurry will set quickly, and the change of flow empty time becomes flat. However, with increase of the water-reducing agent content, the flow time is shortened significantly.

According to the existing form of water in the slurry, the water in the grout can be roughly divided into bound water, free water and wetting water (Pasian et al 2017). After combining with water, cement will interact with each other to generate hydration reactions (Baltazar et al 2014). Chemical reactions will consume the bound water in the slurry. The mixture will not escape from the bound water, and it will not be replaced by water. Wetting water refers to that when cement and fly ash and other cementitious materials in a dry state meets water, a certain amount of water will be adsorbed on the surface of the cementitious materials

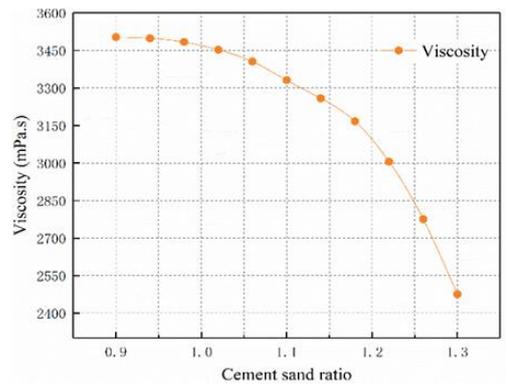
particles, so that the cement and fly ash become wet. Except the combined water and wetting water, all the water remaining in the slurry is free water. Free water can act as a lubricant in the cement mortar. To a large extent, the fluidity of the cement mortar depends on its own lubrication and the content of free water.

When the water-binder ratio increased, the content of free in the cement mortar will also increase, and the fluidity will correspondingly increase. Therefore, the flow time will decrease as the water-binder ratio increases. When the cement-sand ratio is reduced, there is less gelling material between the sand particles, resulting in an increase in frictional resistance between the sand particles, so the fluidity will decrease as the cement-sand ratio decreases, resulting in a longer flow empty time. After adding sodium silicate to the slurry, sodium silicate will chemically react with the calcium hydroxide (Kazemian et al 2011) produced by the hydration reaction to produce a new substance, calcium silicate hydrated, which is a kind of gel and has a certain strength. With the continuous reaction between the two, more and more colloids lead to a decrease in fluidity and a significant increase in empty time. The main chemical component of HS-HF high efficiency water-reducing agent(Yihdego et al 2017) is sodium polymethine naphthalene sulfonate, which is a kind of high molecular active chemical agent, can be adsorbed on the surface of cementitious materials such as cement and fly ash and has an electric field with a negative charge, causing the gel to separate. Therefore, the water reducing agent can greatly improve the fluidity of the cement mortar and reduce the emptying time. Fly ash is a volcanic ash mixing

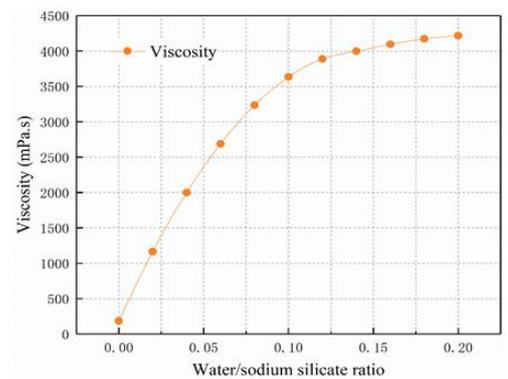
cementitious material with low activity, which can reduce the free water required for chemical reactions. With the addition of fly ash, the free water content increases relatively, thus improving the fluidity of the slurry and reducing the flow empty time.



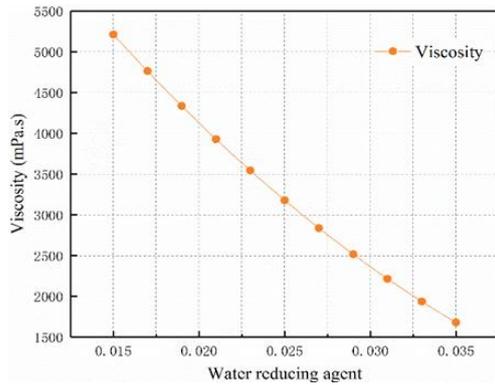
(a)



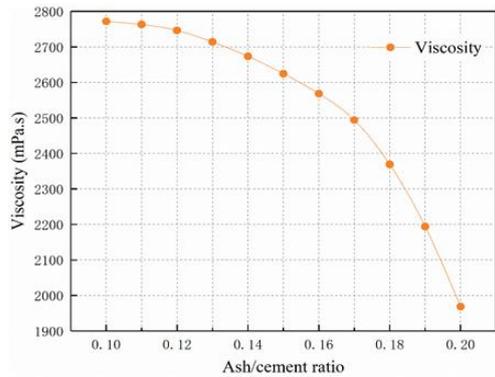
(b)



(c)



(d)



(e)

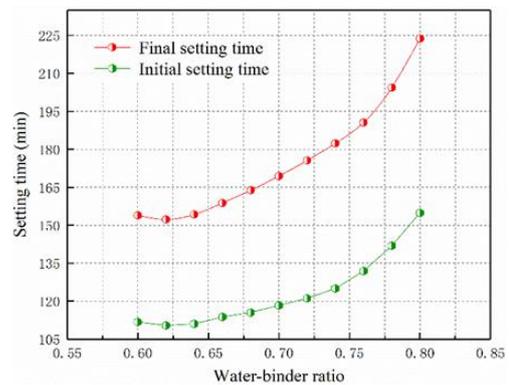
Fig. 3 The relationship between grout viscosity and various influencing factors

Viscosity mainly refers to that during slurry flow, the shear stress that forms a unit velocity gradient in the direction perpendicular to the direction of slurry flow becomes the viscosity of slurry. The grout diffusion radius, grouting pressure and slurry flow have direct influence on viscosity. Viscosity is an important factor to characterize the actual grouting effect.

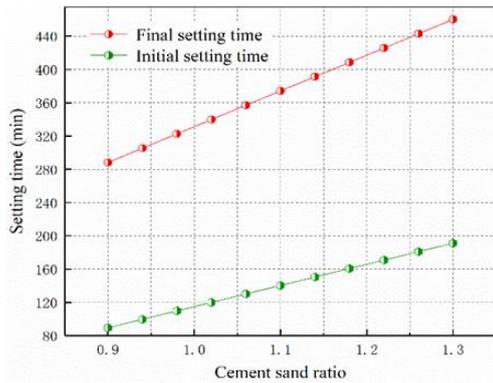
Fig. 3 shows that when the experimental range of each influence factor is given, the size of the slurry viscosity is related to the changes of water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio. The viscosity of slurry was significantly affected by the content of sodium silicate and water reducing agent. The viscosity increases significantly with

increase of sodium silicate. When the content of sodium is very small, the viscosity of slurry can be greatly increased. Viscosity of the slurry decreases significantly with increase of water reducing agent, and the effect of reduction is more significant than the fly ash/cement ratio, water-binder ratio, and cement-sand ratio.

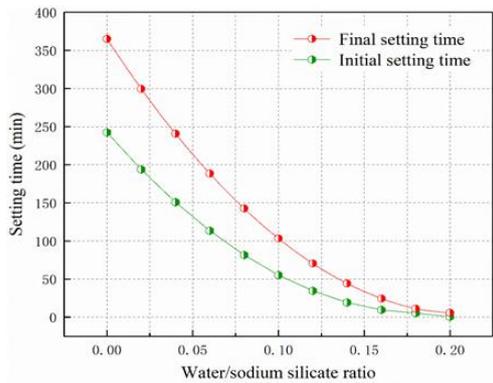
The viscosity of the slurry should not be too large. If the viscosity is too large, it will greatly influence the diffusion range of slurry, making cement mortar in overload, dehydration range too small. So it cannot achieve the ideal effect of grouting, and even making the residual slurry in the grouting pipe too much, which leads to pipe blockage. And The viscosity of the slurry should not be too small. If the viscosity is too small, it will cause that overcome resistance of surrounding rocks will be small, and the setting time is too long and the reinforcement effect is difficult to ensure. Therefore, it is necessary to control the viscosity of the slurry and the dosage of sodium silicate and water reducing agent.



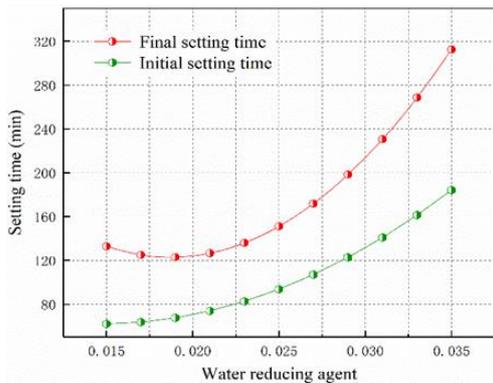
(a)



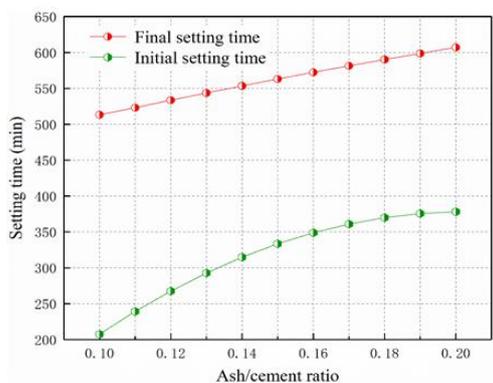
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Fig. 4 The relationship between grout setting

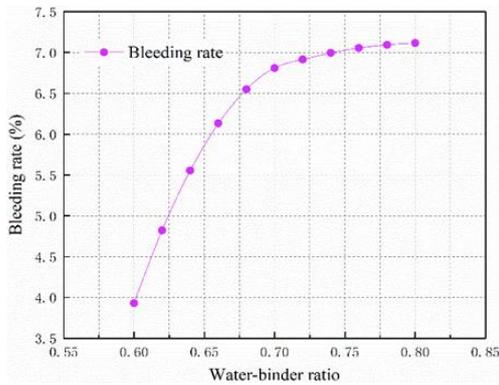
time and various influencing factors

Slurry condensation refers to the process in which after cement, fly ash and other cementitious materials are mixed with water the fluidity and plasticity of slurry gradually disappear over time and form slurry with certain strength. The initial setting time of slurry refers to the time from the addition of water to the beginning of losing plasticity, while the final setting time refers to the time from the addition of water to the complete loss of plasticity. The grout cement hardens is the key factor to form a stone body, and to make the cement mortar form the overall strength.

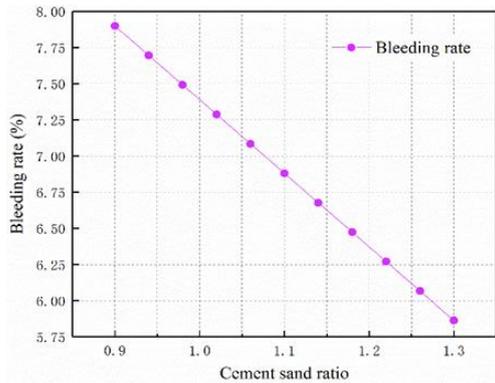
Fig. 4 shows that when the experimental range of each influence factor is given, the length of the slurry setting time is mainly related to the changes of water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio. And the setting time of slurry was significantly affected by the content of sodium silicate and water reducing agent. With the continuous mixing of sodium silicate, the setting time decreases rapidly and presents a nonlinear change. The setting time of slurry can be greatly reduced by adding sodium silicate. With increase of the content of sodium silicate, the slurry will reach rapid setting. The setting time of the slurry was obviously prolonged. With increase of the water-reducing agent, and the effect of reduction is more significant than the fly ash/cement ratio, water/binder ratio, and cement/sand ratio.

The setting time of cement mortar should not be too long. If the setting time is too long, overcome resistance of surrounding rocks will be small, which may lead to some cement mortar loss, and

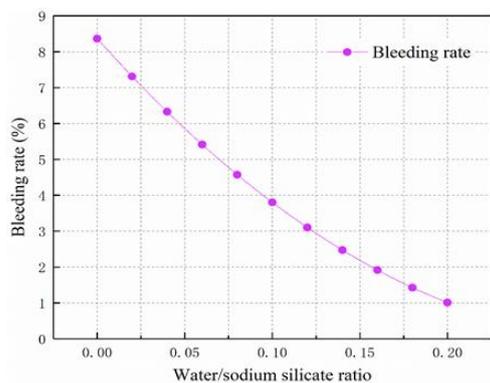
ultimately unable to achieve the ideal grouting effect. The setting time should not be too short. It will cause that overcome resistance of surrounding rocks will be small, and the setting time is too long and the final reinforcement effect is difficult to ensure. Therefore, it is necessary to control the setting time of the slurry reasonably, and grasp the dosage of sodium silicate and water reducing agent.



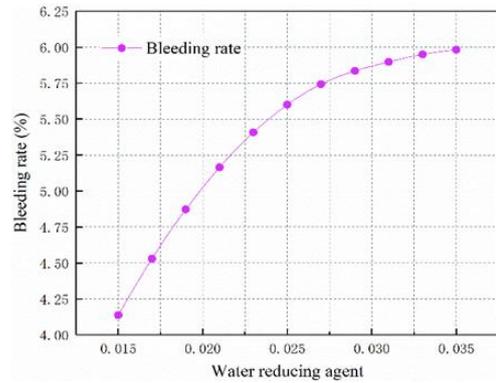
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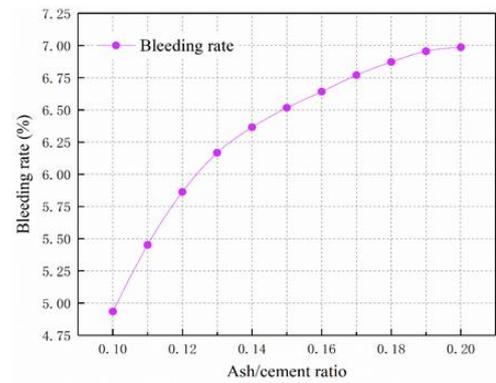
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Fig. 5 The relationship between grout bleeding rate and various influencing factors

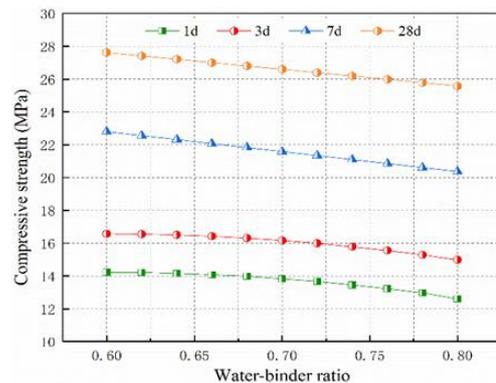
Fig. 5 shows that when the experimental range of each influence factor is given, the amount of water separation from the slurry is related to the changes of water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio. The changes of water/cement ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio have a great influence on the amount of water separation from the slurry. When the content of water reducing agent, water/binder ratio and fly ash/cement ratio increased, the amount of water separation increased obviously, and the bleeding rate also increased and the change trend was positive correlation. With increase of water/sodium silicate ratio and

cement-sand ratio, it decreases, showing a negative correlation trend.

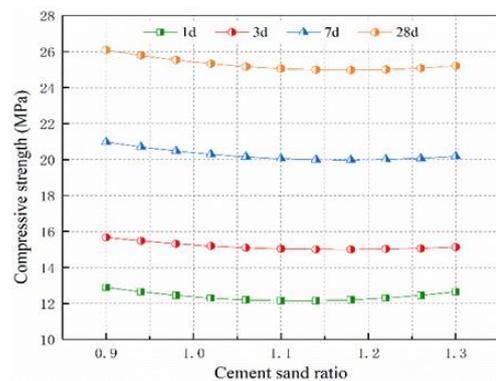
According to the existing form of water in the slurry, cement and fly ash and other cementitious materials in the grouting materials can have affinity interaction with the bound water, and free water can only play a certain lubricating role. With the mixing process, free water can escape from the small pores of the mortar. Among all the base materials for grouting, the density of water is relatively the smallest. When free water escapes from the mixed slurry, it appears the phenomenon of floating up, and the bleeding occurs. If grouting base material grain size distribution is better, the pore is relatively thin, various material accumulation is denser, then free water needs to take very long to escape from the slurry. Cement and other cementitious materials will consolidate along with the extending of time, block the escape way to free water, and slurry finally precipitate free water will have little or no. In the case of a certain amount of water, increasing the amount of cementitious materials will lead to an increase in the total specific surface area of the cement mortar mixture. In the case of only a certain amount of water, more surface of the cementitious materials will be wetted, so the free water precipitated from the slurry is greatly reduced. Similarly, when the cement-sand ratio increases, the amount of emery is relatively reduced, and the amount of cementitious material increases. Cement and fly ash, which are thinner than emery, will refine the pores in the cement mortar, reduce the connectivity between the pores, and lead to the reduction of bleeding yield.

The main chemical component of HS-HF high efficiency water-reducing agent

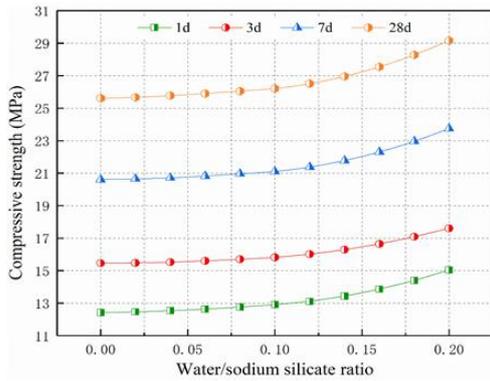
is sodium polymethine naphthalene sulfonate, which is a kind of high molecular active chemical agent, can be adsorbed on the surface of cementitious materials such as cement and fly ash, and can produce a negative electric field. This kind of electric field with a negative charge causes the gel particles to repel and separate from each other, so the coagulation is reduced, the required free water for wetting is reduced, and the amount of free water precipitated from the slurry is increased. Because the activity of fly ash is lower than that of cement, increasing the amount of fly ash causes the bound water in the cement mortar to be significantly reduced, which leads to bleeding increase.



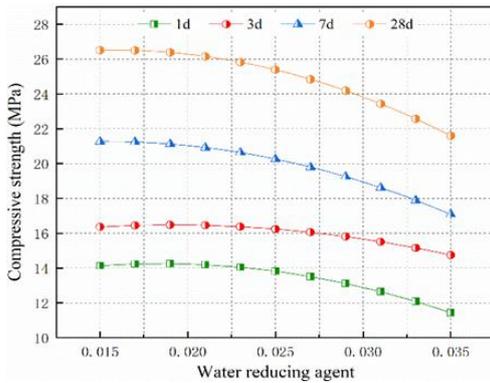
(a)



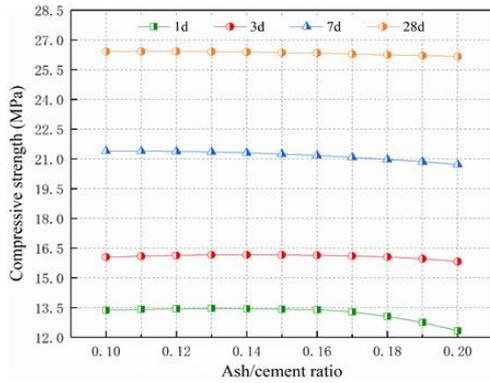
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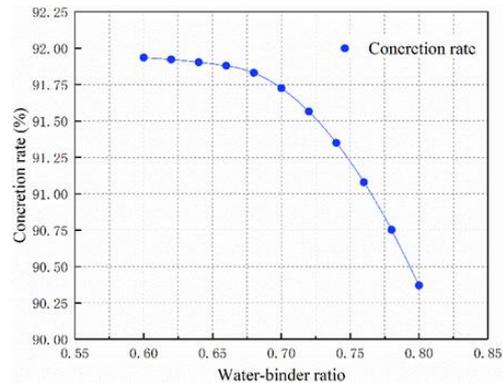
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Fig. 6 Relationship between compressive strength and various influencing factors

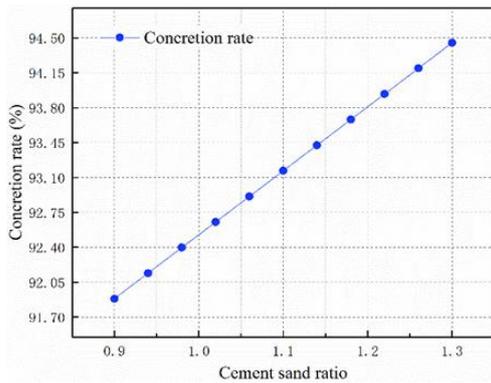
Fig. 6 shows that when the experimental range of each influence factor is given, the compressive strength of the slurry increases gradually over time, and the compressive strength of the slurry increases significantly in the first 7 days, and then increases slowly.

From the above five figures, it can be concluded that, within the given test value

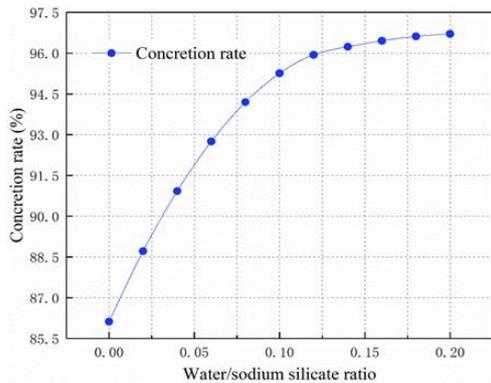
range of each factor, the compressive strength of the slurry is related to the change of water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio. And the change of the cement/sand ratio and water/sodium silicate ratio have a significant impact on the compressive strength of the concretion. With increase of fly ash/cement ratio and water-reducing agent content, the compressive strength from day1 to day28 increased significantly, showing a positive correlation. With increase of water-cement ratio, water-water/sodium silicate ratio and cement-sand ratio, it decreases with a negative correlation trend. It is necessary to reasonably regulate the water/cement ratio, cement/sand ratio, water/sodium silicate content, water reducing agent content, and fly ash/cement ratio of the slurry, so as to improve the strength gel in the early stage as far as possible, effectively improve the surrounding rock environment and control the deformation.



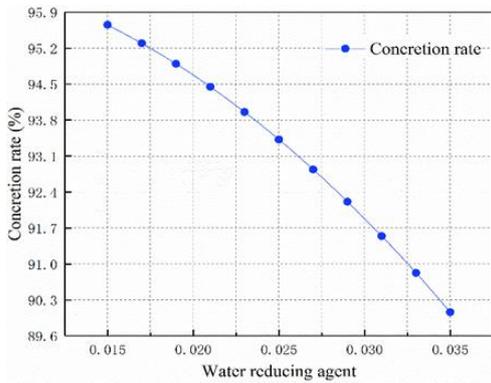
(a)



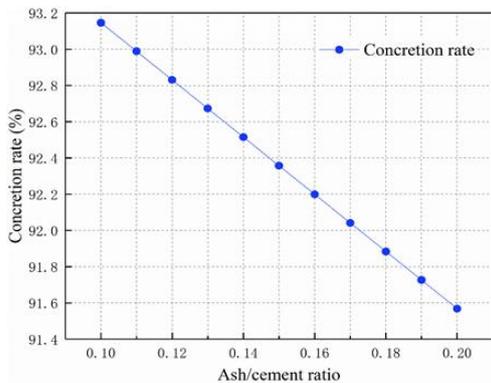
(b)



(c)



(d)



(e)

Fig. 7 The relationship between grout concretion

rate and various influencing factors

Fig. 7 shows that when the experimental range of each influence factor is given, the setting rate of cement mortar is related to the change of the water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio. With the change of the content of water reducing agent, water/cement ratio and water/sodium silicate ratio, the concretion rate of cement mortar changes remarkably. The concretion rate of grouting material will decrease. With increase of water-reducing agent content, water-cement ratio and fly ash/cement ratio, showing a negative correlation. The increase of sodium silicate content will significantly increase the concretion rate of cement mortar. When the content of sodium silicate increases to 1/5, the concretion rate can reach 100%, with a positive correlation trend.

Starting from the initial setting to the front of the concretion time, under the action of pressure, the cement mortar is dehydration density, the free water in the cement mortar is increased, and the slurry consolidation rate is reduced. The increase in the content of the water reducing agent and the increase in the amount of water make the free water in the slurry increase, and sodium silicate will significantly reduce free water, resulting in a greater slurry concretion rate. The activity of fly ash is lower than that of cement, which will cause the bound water in the cement mortar to decrease and the free water to increase. As the fly ash ratio increases, the setting rate decreases, but the overall fly ash/cement ratio has little effect.

Based on the analysis of the influence of the above-mentioned single factors on the

cement mortar, the conclusion of the significant influence of each experimental

factor on the basic properties of grouting material was obtained, as shown in table 6:

Table 6 Test Factor Significance Analysis Table

Experimental parameters	Water/binder ratio	Cement/sand ratio	Water/sodium silicate ratio	Water reducing agent	Fly ash/cement ratio
fluidity	not significant	significant	significant	not significant	not significant
Viscosity	not significant	not significant	significant	significant	not significant
setting time	not significant	not significant	significant	significant	not significant
bleeding rate	significant	not significant	significant	significant	significant
compressive strength	not significant	significant	significant	not significant	not significant
concretion rate	significant	not significant	significant	significant	not significant

Based on Table 6 and the comparative analysis of the single-factor relationship curve, it can be seen that the effect of water-binder ratio on the concretion rate and the bleeding rate of the grouting material grout is more significant than on the fluidity, viscosity, setting time and compressive strength. Considering the effect and performance of the actual engineering grouting, the water/binder ratio is preferably selected to be 0.75. The cement/sand ratio factor has only a significant impact on compressive strength and fluidity, and considering the economy of different levels, the ratio of cement/sand ratio is 1.2. The content of sodium silicate has a significant impact on the technical indicators of fluidity, viscosity, setting time, compressive strength, concretion rate and bleeding rate. Considering that the geology of the roadway in Hongyan Coal Mine is soft rock and the inject ability of grouting material, the preferred level of water/sodium silicate ratio is 8%. Water reducing agent has a significant impact on viscosity, bleeding rate and setting rate. After the Water reducing

agent is added, the amount of cement can be greatly reduced, and the cost is reduced. The level of priority of the water reducing agent is 3%. Because the fly ash/cement ratio has a more significant effect on the bleeding rate than other influencing factors, combining the grouting performance and pipeline transportation, the pipe blockage phenomenon can be avoided. The level of the fly ash/cement ratio is preferably 0.18. In summary, through systematical analysis of the technical parameters of the slurry with different mixture ratios, combined with the geological conditions and support costs of the soft rock roadway in Hongyan Coal Mine, the optimal component quality percentage of the impact factor of grouting materials were water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water reducing agent and fly ash/cement ratio was 0.75, 1.2, 8%, 3% and 0.18. After conversion, the optimization of the grouting material is shown in table 7. We have listed some comparisons regarding various performance indexes of the grouting material in the following table 8. By

comparison, the novelty and the significance of our paper are highlighted.

Table 7 Optimal mixture ratio of grouting material

Material	Cement	Fly-ash	Sodium silicate	Emery	Water	Water reducing agent
Content (kg/m ³)	423.7	76.3	30	416.7	375	15

Table 8 Performance indexes of the grouting material comparisons of some major and representative grouting material studies with soft rock considered

Indices	This paper	Hideki et al. (2014)	Sun, et al (2017)	Zhang et al. (2018)
Applicable conditions	Broken soft rock Weathering Spalling Swelling	High strata stress Weak floors	Preventing swelling of soft rock Broken	sealing groundwater inflow reinforcing wall rock in deep mine
Compressive strength (MPa)	28.5	/	21.5	0.52
Viscosity (mPa.S)	70	40	90	/
Setting time (h)	1/2	/	5.8	5/6
Bleeding rate (%)	0	0	0	15
Permeability coefficient (cm/s)	5×10^{-3}	/	/	/
Cast (CNY/m ³)	349	/	/	/
Field grouting effect	The integrity and self-stability of the surrounding rock have been fully improved	It is reasonably accurate when the flow of the grouting material is laminar	It has good reinforcement effect	It effectively filled the fractures and joint planes of the surrounding rock

The new composite grouting material used in this paper has a rich resource and lower cost, which greatly reduces the cost of grouting. Table 9 is the cost comparison of new composite grouting material and No-modificated cement grouting.

Table 9 Comparison cost of the new composite grouting material with the cement grouting material

Slurry species	Grout formula	1m ³ Slurry raw materials amount (kg)	Unit price (CNY/t)	1m ³ slurry cost (CNY)
New composite grouting material	C: F: B: S: W: A =1.13:0.21:0.08:1.1 1:1:0.04	Cement 423.7 Fly ash 79.3 Sodium silicate 30 Emery 416.7 Water 375 Water reducing agent 15	250 60 500 400 5 3600	349
Cement grouting material	W/C=1.0	Cement 1600 Water 500	250 5	403

4 Field experiment of the bolt-grouting effects

The surrounding rock of Hongyan Coal Mine has a high degree of broken rock and a large potential risk coefficient. A good effect needs to be achieved in a short period of time. Therefore, the grouting material developed in this paper was used to grout and reinforce the broken surrounding rock of 10103 tailgate in Hongyan coal mine as shown in Fig. 8(a), and the grouting effect was monitored by using borehole camera. The result is shown in the Fig. 8(b). It shows that before grouting, the surrounding rock was broken and there were many joints, fissures and cracks. After grouting, the grout effectively filled the cracks of the surrounding rock and reinforced the surrounding rock of the roadway.



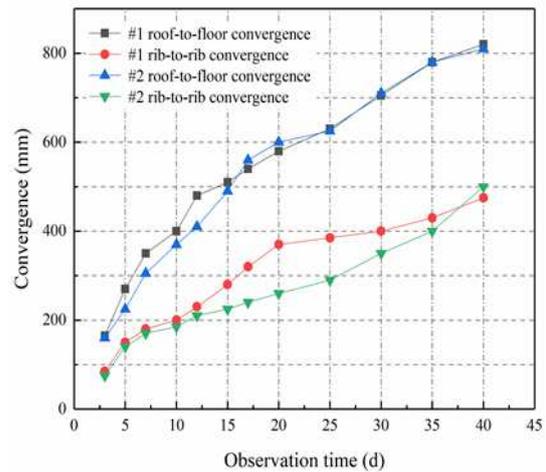
(a)



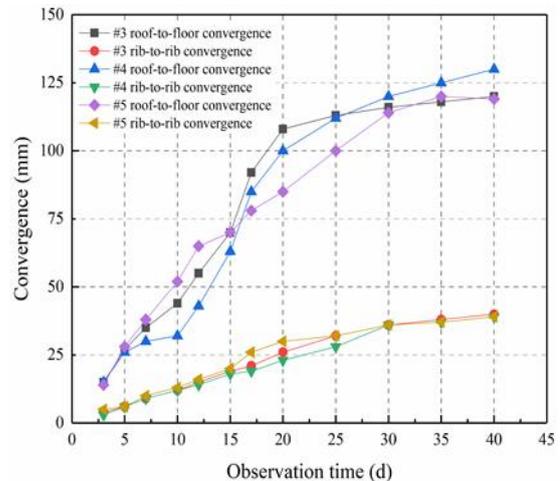
(b)

Fig. 8 Borehole camera images of surrounding rock in the roof and ribs

At the same time, the deformation of surrounding rock before and after grouting in 10,103 tailgate was instrumented. The displacement curve is shown in the Fig. 9, and the instrumentation plan is shown in the Fig. 10.



(a)



(b)

Fig.9 Convergence of 10103 tailgate monitoring diagram

As shown in Fig. 9, after 40 days, the maximum roof-to-floor convergences of 10,103 tailgate grouting treatment were 129.3 mm, and the maximum rib-to-rib convergence of 10,103 tailgate grouting treatment were 39.7 mm. Compared with the non-grouting area, the value has a significant decrease. It shows that the

grouting material effectively entered the weak surface of the roof and roadway ribs, the integrity and self-stability of the surrounding rock were improved, and it can

resist large in-situ stress and secondary engineering disturbed stress. Grouting played a significant role in controlling the deformation of surrounding rock.

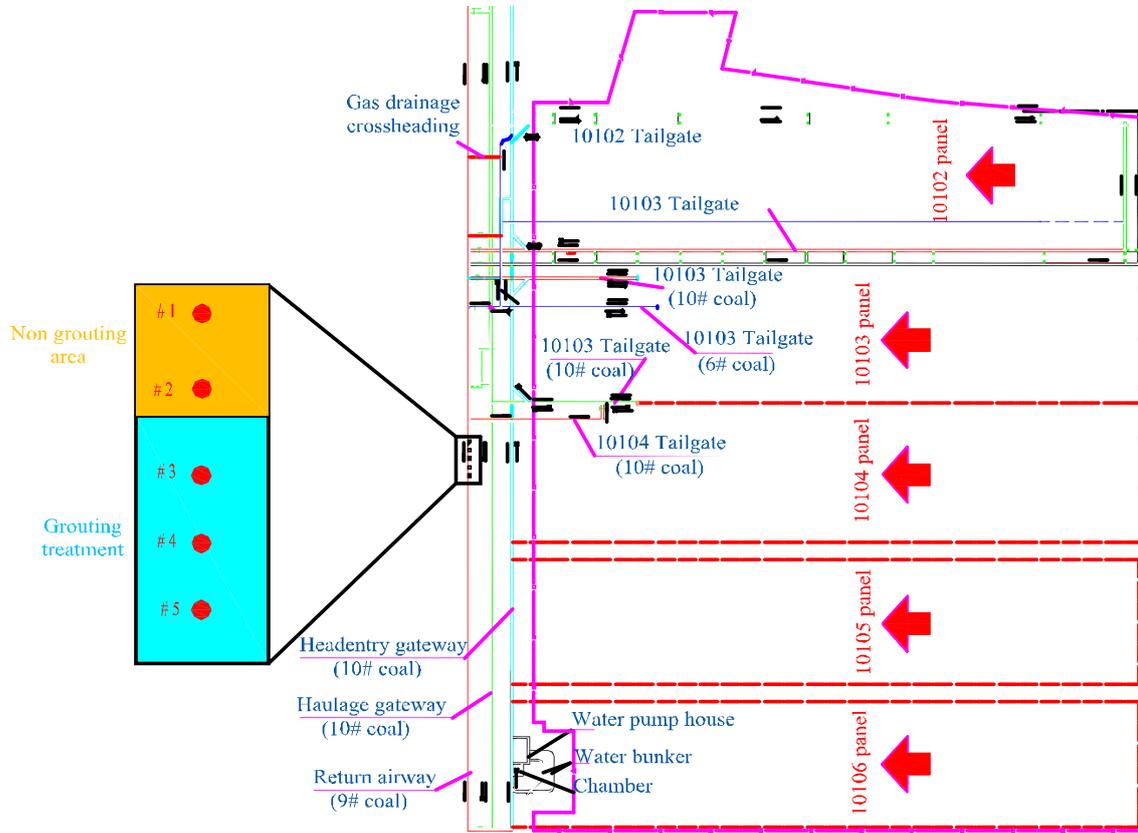


Fig. 10 Instrumentation plan of displacement meters (localized enlarged views, not to scale)

5 Conclusions

(1) With increase in water/binder ratio, fluidity, setting time, slurry bleeding rate and compressive strength of concretion increase, viscosity and concretion rate decrease. With increase in cement/sand ratio, fluidity, setting time and concretion rate increase, viscosity, bleeding rate of slurry and compressive strength of concretion decrease. With increase in sodium silicate, viscosity, concretion rate and compressive strength of concretion increase, fluidity, setting time and bleeding rate of slurry decrease.

(2) With increase in water reducing agent, fluidity, setting time and slurry bleeding rate increase, but viscosity, concretion rate and compressive strength of concretion decrease; As fly ash/cement ratio increases, fluidity, setting time and slurry bleeding rate increase, but viscosity stone rate, concretion rate and compressive strength of concretion decrease.

(3) Water/sodium silicate ratio and cement-sand ratio have a significant effect on fluidity of slurry and compressive strength of concretion. Effect of sodium silicate and water reducing agent on viscosity and setting time of slurry is more

significant. Fly ash/cement ratio, water/sodium silicate ratio and water reducing agent have a more significant effect on bleeding rate of slurry. Water/binder ratio, water/sodium silicate ratio and water reducing agent have a greater effect on concretion rate of slurry.

(4) After analyzing the various technical indexes of slurry with different mixture ratios, combined with geological conditions and support costs of soft rock roadways in Hongyan Coal Mine, an optimal component percentage of each contributing factor of the grouting material was determined as the water/binder ratio, cement/sand ratio, water/sodium silicate ratio, water-reducing agent and fly ash/cement ratio was 0.75, 1.2, 8%, 3% and 0.18, respectively.

(5) The engineering application results verify that its performance meets the needs of grouting treatment for weak and broken rock mass, and its performance is better than traditional cement materials. At the same time, the new composite grouting materials make full use of industrial waste and emery, reducing material costs and significantly improving engineering economy.

Acknowledgements

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Figures

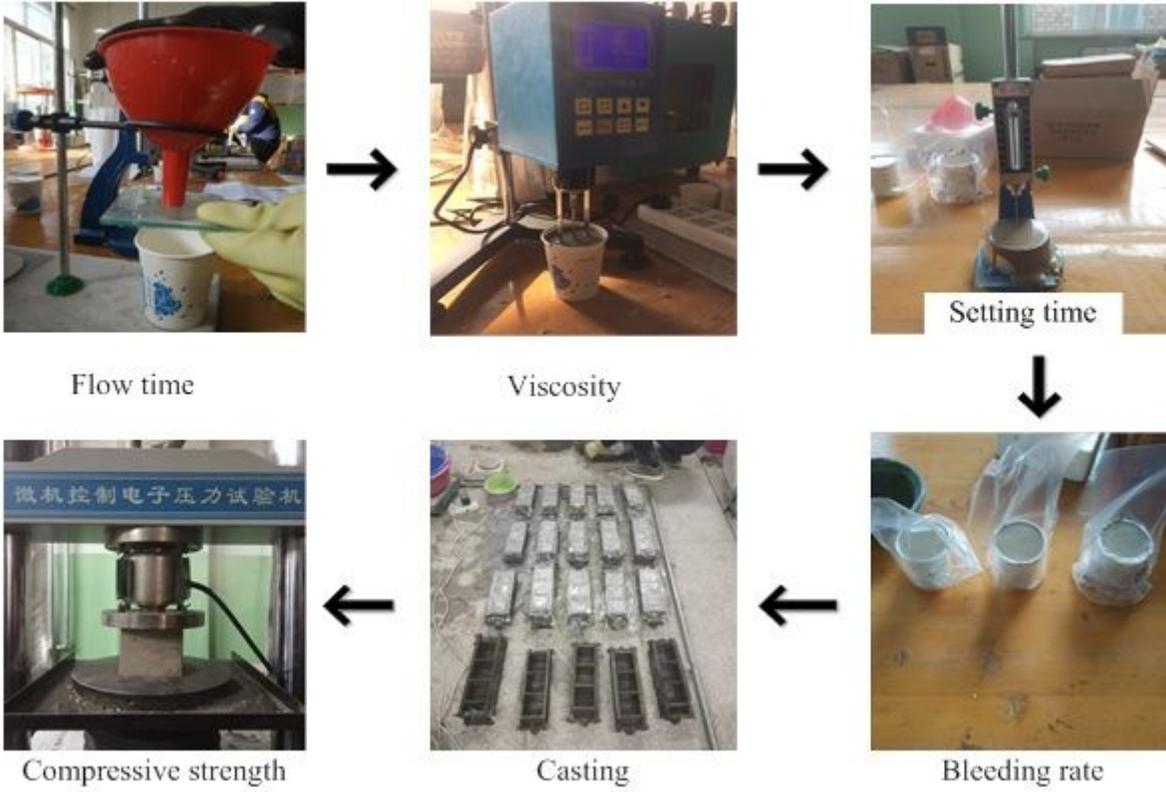
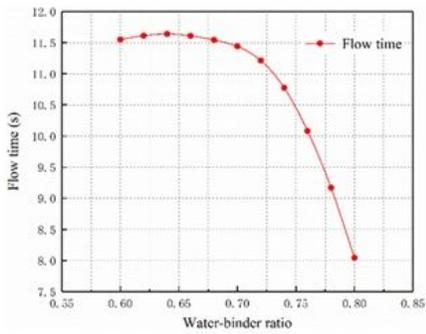
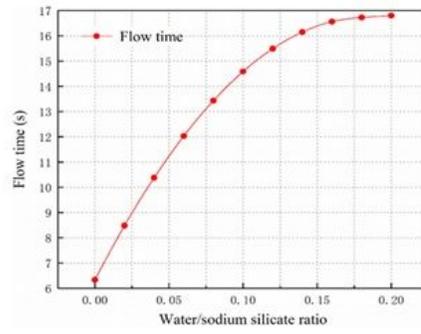


Figure 1

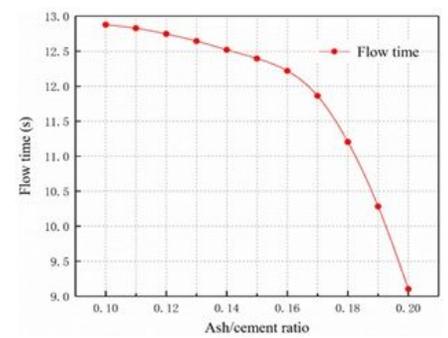
Test steps



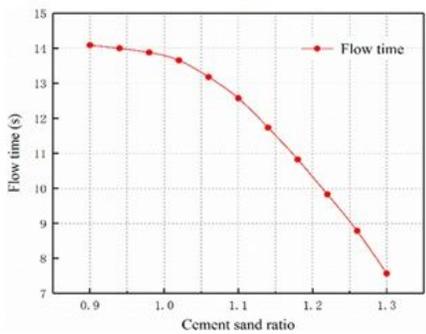
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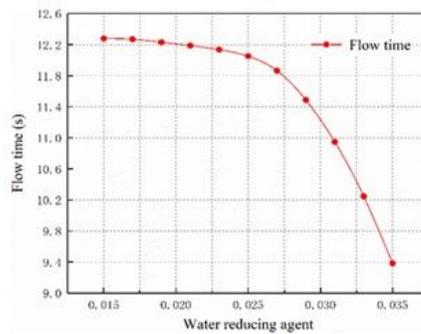
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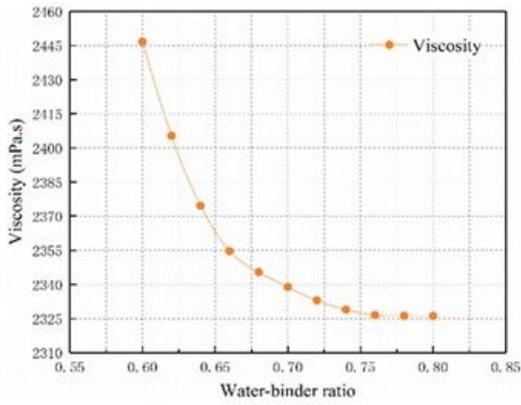
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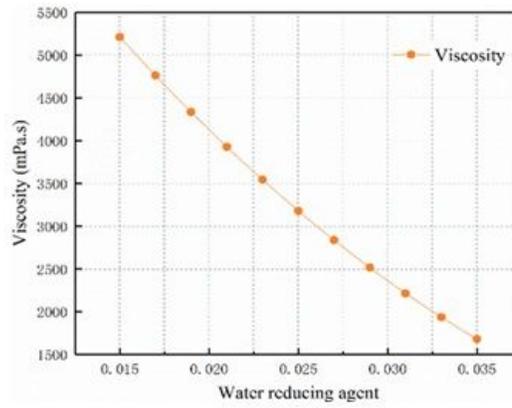
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Figure 2

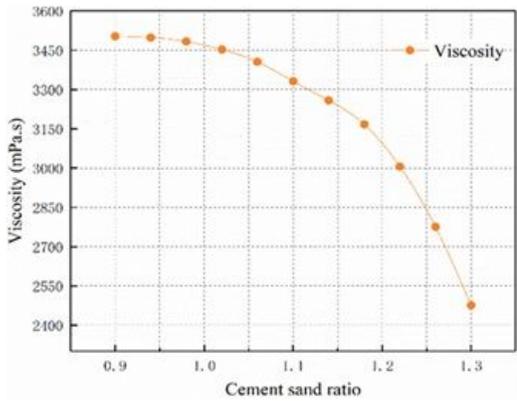
The relationship between grout flow time and various influencing factors



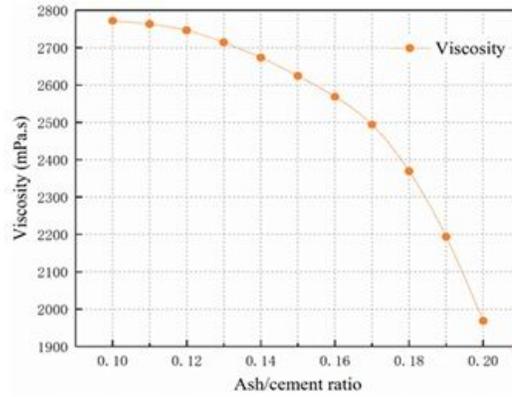
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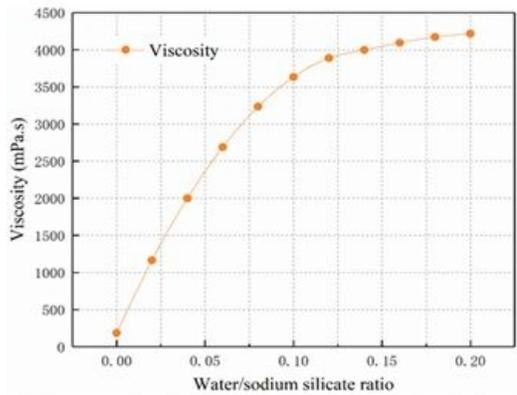
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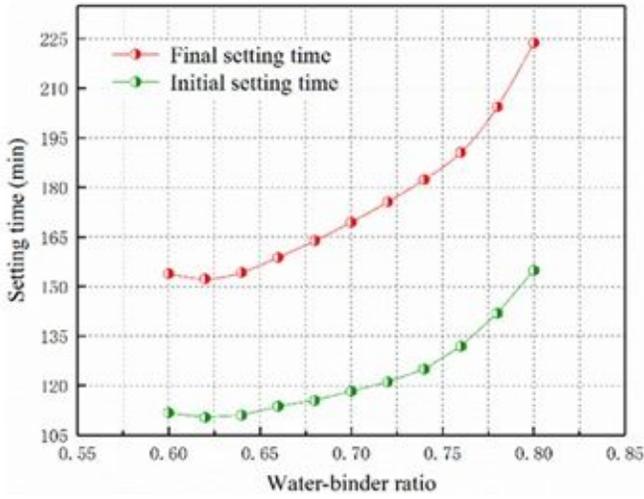
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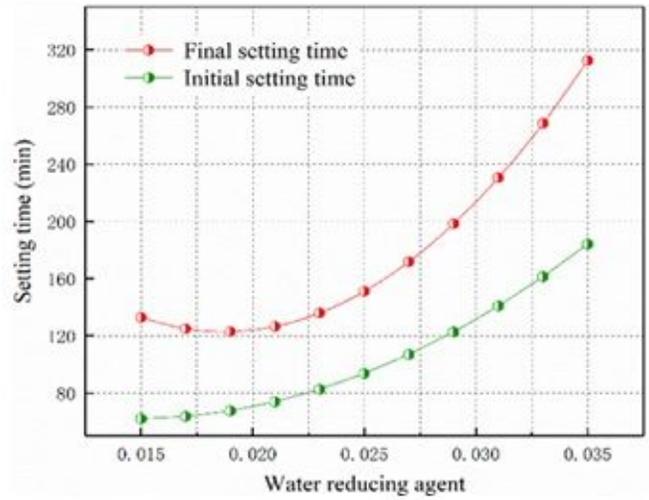
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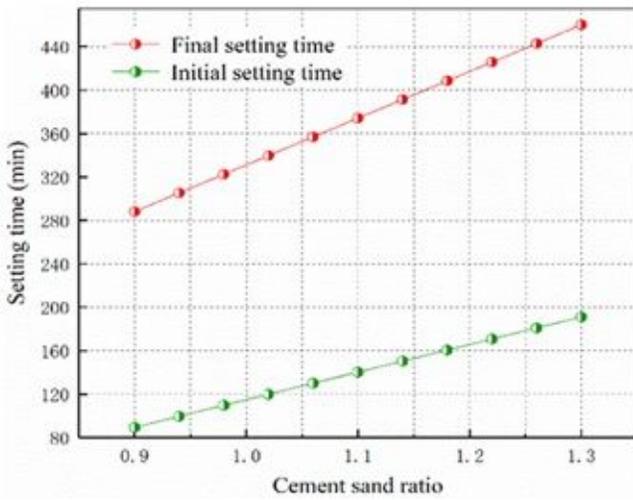
The relationship between grout viscosity and various influencing factors



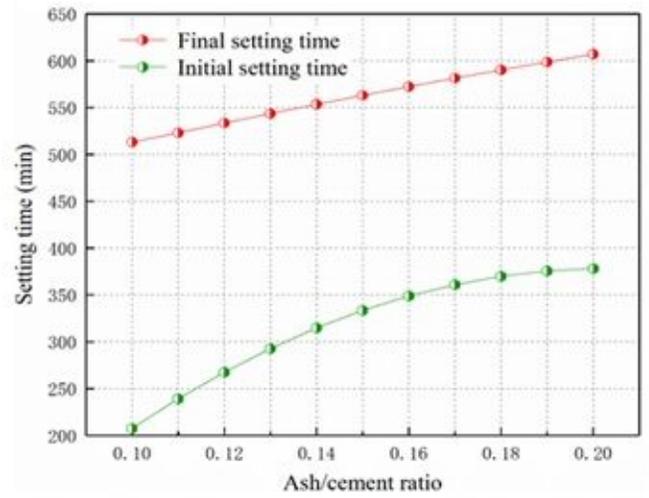
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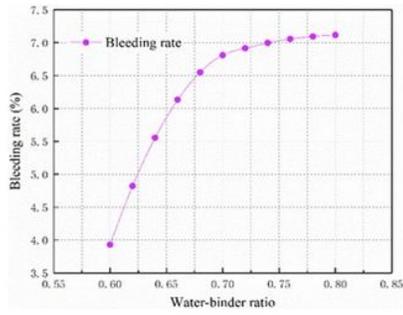
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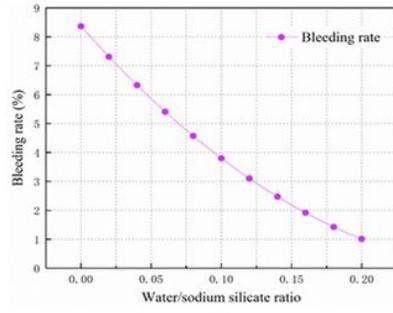
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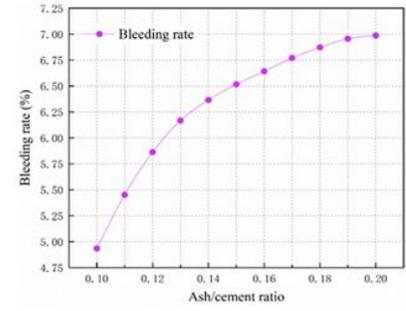
The relationship between grout setting



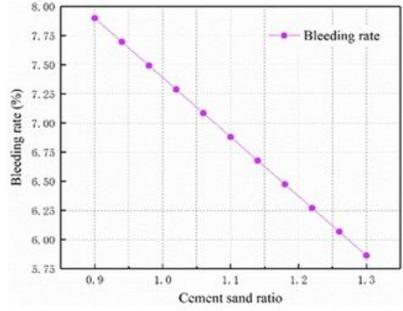
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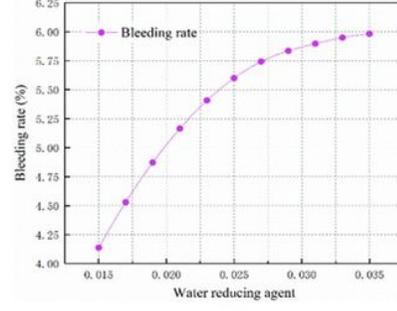
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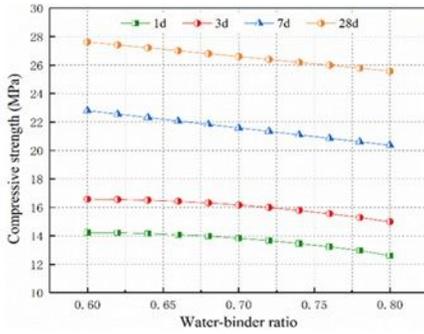
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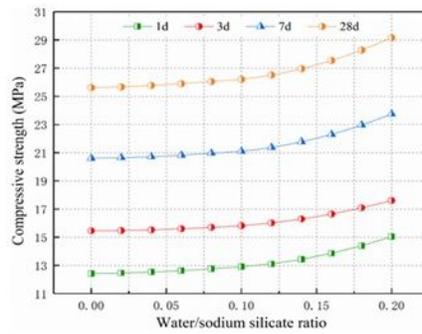
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Figure 5

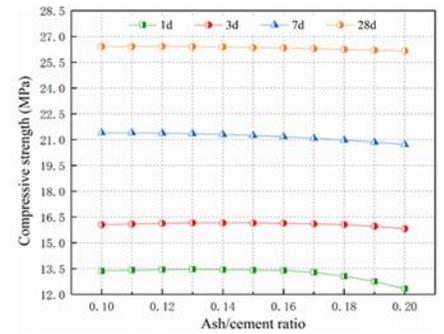
The relationship between grout bleeding rate and various influencing factors



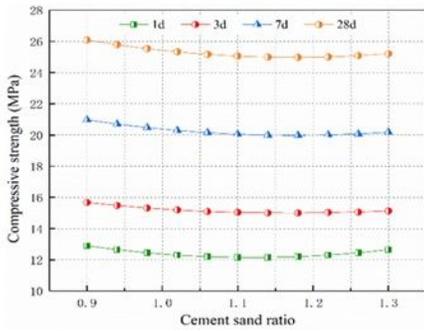
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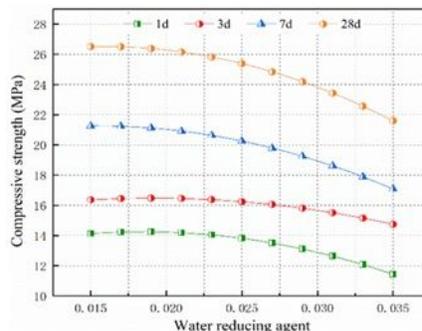
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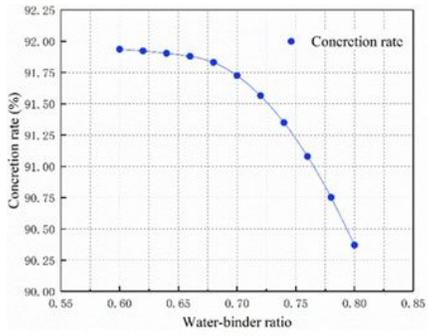
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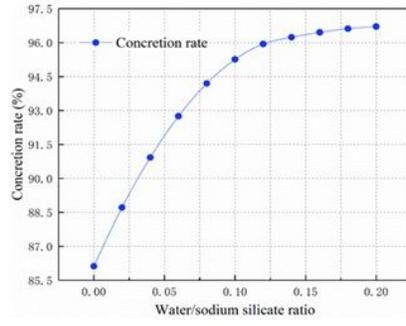
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Figure 6

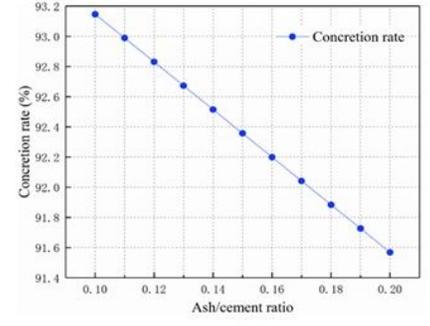
Relationship between compressive strength and various influencing factors



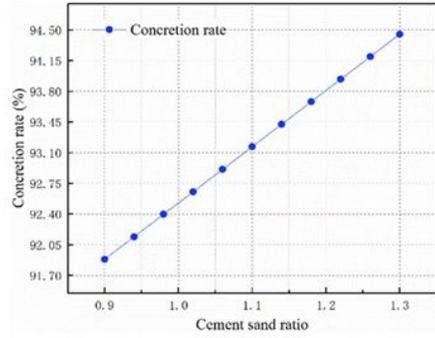
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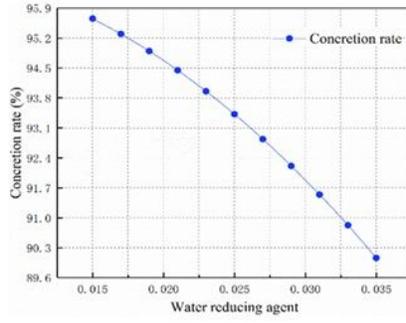
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(e)



(b)



(d)

Figure 7

The relationship between grout concretion rate and various influencing factors



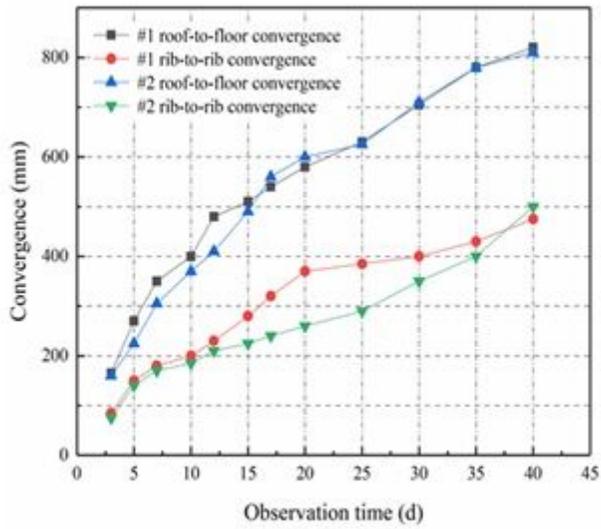
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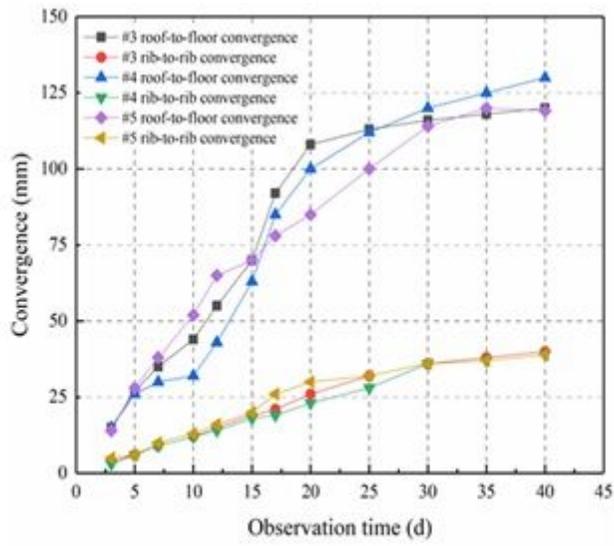
(b)

Figure 8

Borehole camera images of surrounding rock in the roof and ribs



(a)



(b)

Figure 9

Convergence of 10103 tailgate monitoring diagram

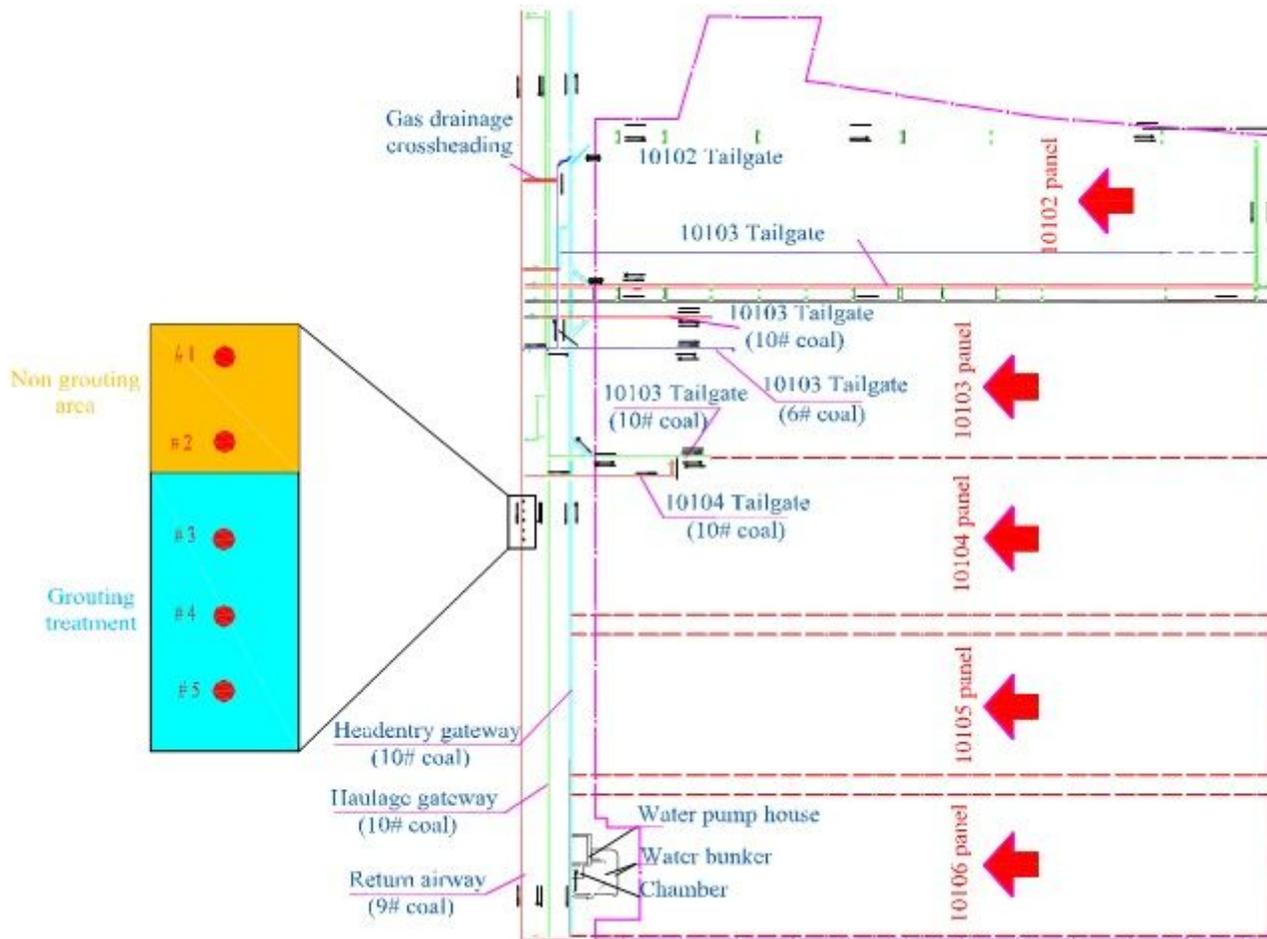


Figure 10

Instrumentation plan of displacement meters (localized enlarged views, not to scale)