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RESEARCH ARTICLE

Selective Tests of Grouting Material for Fractured Coal and Rock Masses in a Water-borne H_2S Environment

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Abstract: Coal mining is vulnerable to multiple kinds of threat from water incursion. Many coal seams contain dissolved H_2S, which may be released during mining, seriously endangering the health of workers. Orthogonal testing was used to analyze the physical and mechanical properties of composite slurries in different proportions. The results showed slurry with a water-solid ratio of 0.8:1 to have the optimal combination of properties. A uniform experimental method was used to investigate the impact of the water-cement ratio, concentration of sodium silicate, and volume of cement-sodium silicate (CS) on the setting time and consolidating strength of CS slurry. This paper provides the appropriate application scope of two grouting materials (optimized composite slurry and CS slurry), based on experimental data and the results of a large number of *in situ* trials. Finally, the optimized composite slurry and cement-sodium silicate slurry were used to carry out grouting of the center auxiliary transport roadway of the 3^{-1} coal seam at the Ningtiaota Coal Mine, operated by the Shaanxi Coal Industry Group. It was shown that the type and formula of the slurry could be changed in a timely manner, based on the water outlet and concentration of H_2S at the site, achieving successful grouting reinforcement and seepage-proofing.

Keywords: Water-borne H_2S environment, Fractured coal and rock masses, Composite slurry, Optimized design, Cement-sodium silicate slurry, Optimization formula test, Engineering application.

1. INTRODUCTION

The coal fields of China vary widely in terms of the age and nature of the seams, and many exhibit complex geology. In particular, many mines that are threatened by unpredictable water intrusion also contain H_2S. This presents a serious safety hazard for those working in the shaft. Grouting engineering is therefore an important technology to prevent flooding of mines [1 - 6].

Traditional slurry, including pure cement slurry, cement-fly ash slurry, cement-clay slurry, and chemical slurry, all have drawbacks, and attention is now turning to grouts of composite type. Approaches to improving the strength of grouting materials have included the use of composite cement-sodium silicate, proposed by Maoyin Wu *et al*. [7]; sodium silicate with high Baume degree or composite cement grouting with the appropriate ratio of cement, fly ash and mineral powder can increase the strength of slurry concretion. Wang *et al*. [8] conducted performance testing and analysis of a new composite slurry with respect to fluidity, water outlet time, initial and final setting time, concretion strength and permeability. It was demonstrated that neither of above two slurries was suitable for use in environment with a significant presence of H_2S, and nor did they contribute to decreasing the cost of grouting.

Cost considerations are very important in the selection of grouting materials. Fly ash, an industrial by-product, has the same grain size and distribution as cement, but with more spheroidal particles. The addition of fly ash to cement slurry may both reduce the cost and improve the fluidity of the slurry. However, adding an excessive amount will lower the initial strength of the cement slurry. We have shown that the slurry of P.O32.5 cement in a 1:1 ratio of water

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to cement has a bending strength of 0.73MPa after three days, with a compressive strength of 3.14MPa. After the addition of 40% fly ash, the bending strength after three days fell to 0.30MPa, with a 55% reduction, and the compressive strength fell to 2.10MPa, by a 31% reduction. From the previous literature, it is observed that calcined lime and sodium sulfate are able to effectively arouse the activity of fly ash, improving the early concretion strength of cement-fly ash slurry. Calcined lime is also able to reduce the concentration of H_2S. In this study, therefore, cement and fly ash were used to form composite slurry, and the activating agents (calcined lime and sodium sulfate) and clay were also used to optimize the performance. Cement-sodium silicate slurry uses the addition of sodium silicate to improve the setting time, and is widely used in coal mining and other applications requiring rock mass dynamic grouting, however, the slurry is more expensive, and is not able to reduce the H_2S concentration effectively.

In this study, the ratio and characteristics of composite slurry comprising cement, fly ash, calcined lime, sodium sulfate, and clay have been analyzed. The results will allow operators to adjust the ratios in the two grouting materials discussed above in real time to achieve the optimal grouting effectively, based on the water inflow to the coal and rock mass and the concentration of H_2S.

2. PREPARATION OF COMPOSITE SLURRY

2.1. Experiment Principles and Design

2.1.1. Basic Principles of Activating Agent

The Ca^{2+} of quicklime was provided the active particles for fly ash, making the SiO_2 in the fly ash creating a Calcium-Silicate-Hydrate (CSH) gel. Active Al_2O_3 cannot generate CAH under the independent action of Ca^{2+} and can generate by combining action of Ca^{2+} and SO_4^{2-}. SO_4^{2-}, reacting with the Ca^{2+} to generate CSH, speeding up the hydration of the fly ash and improving its strength.

2.1.2. Experimental Method

① Composition of slurry

The slurry material comprised P.O 32.5 ordinary Portland cement, fly ash (from the Xi'an Baqiao Power Plant), clay, quicklime with CaO concentration no less than 98%, Na_2SO_4 with the concentration no less than 99%, and water.

② Experimental design

The composition of the slurry was determined based on previous studies and indoor trials. Because the experiment had many factors and levels, an orthogonal design of $L_{16}(4^5)$ was adopted, and 16 experiments were conducted. The experimental factors were: the water-solid ratio (with levels 0.5**:**1, 0.8**:**1, 1.0**:**1, and 1.5**:**1), the mass percentage of fly ash in the cement (15%, 25%, 35%, and 45%), the mass percentage of clay in the cement (10%, 15%, 20%, 25%), the mass percentage of quicklime CaO in the cement (2%, 3%, 5%, and 7%), and the mass percentage of Na_2SO_4 in the cement (1%, 3%, 5%, and 7%), where the water-solid ratio was that between water and the total mass of cement, fly ash, and clay.

2.2. Experimental Results

2.2.1. Physical Properties of Slurry

The experimental results are shown in Table **1**. It can be seen that, in terms of stability, the slurry with water-solid ratios of 0.5**:**1 and 0.8**:**1 had a water-liberating rate less than 5%, marking them as stable. The slurries with water-solid ratios of 0.5**:**1 and 0.8**:**1 had concretion rates greater than 90%, which is relatively high. The slurry with a water-solid ratio of 0.5:1 had the highest viscosity, being almost no-flow. Slurry for use in engineering applications must have liquidity to allow pumping, so that slurry with this water-solid ratio is only suitable for special applications. These results therefore identified the slurry with a water-solid ratio of 0.8:1 as having the optimal physical properties.

2.2.2. Mechanical Properties of Slurry Concretion

The bending strength and compressive strength of the non-lateral confinement of the slurry concretion was next tested (Fig. **1**), using a DKZ-5000 Type Power-Driven Bending Testing Machine (Fig. **2**) and NYL-300C Type Compressive Testing Machine (Fig. **3**) manufactured by Wuxi Building Material Instrument And Machinery Co., Ltd.

(Table **2**) shows the test results. As can be seen, the bending strength and unconfined compressive strength of the composite slurry of different proportions increased at different rates. As an example, the increase in bending strength and unconfined compressive strength over time of the composite slurry concretion from the 3rd group is shown in Fig. (**4**).

Table 1. Experimental result of physical properties of composite slurry.

Experimental group	Water-solid ratio	Mass percentage of fly ash in the cement (%)	Mass percentage of clay in the cement (%)	Mass percentage of quicklime in the cement (%)	Mass percentage of Na_2SO_4 in the cement (%)	Initial maxing viscosity(s)	Bulk density (G/cm^3)	Concretion ratio (%)	Water-liberating rate (%)	Setting time(h:min)	
	A	B	C	D	E					Initial setting	Final setting
1	0.5:1	25	20	3	5	-	1.80	99.3	0.5	6:35	12:25
2	1.0:1	45	10	3	3	22	1.43	87.5	13.0	20:45	31:15
3	0.8:1	45	20	5	7	31	1.53	93.3	4.2	13:15	23:05
4	1.5:1	25	10	5	1	20	1.32	71.8	30.5	31:15	48:35
5	0.5:1	35	10	7	7	-	1.80	98.3	1.0	7:0	12:15
6	1.0:1	15	20	7	1	25	1.44	86.3	7.0	19:35	30:05
7	0.8:1	15	10	2	5	32	1.58	95.0	4.2	11:45	20:55
8	1.5:1	35	20	2	3	18	1.32	66.3	43.6	31:05	48:10
9	0.5:1	15	25	5	3	-	1.80	99.5	0.2	7:30	12:50
10	1.0:1	35	15	5	5	23	1.45	88.3	15.1	19:05	29:15
11	0.8:1	35	25	3	1	29	1.50	93.3	3.1	12:30	21:45
12	1.5:1	15	15	3	7	20	1.32	68.8	37.4	30:45	47:55
13	0.5:1	45	15	2	1	-	1.79	98.8	1.0	7:35	13:10
14	1.0:1	25	25	2	7	24	1.43	91.3	5.1	20:15	31:05
15	0.8:1	25	15	7	3	33	1.56	93.3	2.5	12:05	21:15
16	1.5:1	45	25	7	5	18	1.34	72.0	30.5	31:25	49:10

Fig. (1). Test sample.

Fig. (2). Electric bending testing machine of DKZ-5000.

Fig. (3). Compressive testing machine of NYL-300C.

Table 2. Experimental results of mechanical properties of composite slurry.

Experimental group	Bending strength (MPa)				Unconfined compressive strength (MPa)			
	3d	**7d**	**14d**	**28d**	**3d**	**7d**	**14d**	**28d**
1	1.99	2.34	4.15	5.39	9.51	12.34	16.75	21.38
2	0.48	0.75	0.81	1.11	1.50	2.82	3.31	4.32
3	0.65	1.11	1.60	2.21	2.48	3.85	5.03	8.18
4	0.29	0.30	0.70	1.13	0.96	1.43	2.61	3.57
5	1.64	2.08	3.88	5.01	7.36	10.02	12.66	19.81
6	0.31	0.66	1.81	2.51	2.24	2.36	3.14	4.52
7	0.97	1.49	2.00	2.63	4.28	4.74	7.36	9.28
8	0.32	0.60	0.81	1.22	1.74	2.31	3.16	4.19
9	2.01	2.25	3.99	5.18	8.38	11.78	16.03	21.44
10	0.56	0.82	1.01	2.20	2.25	4.37	6.02	8.32
11	0.31	0.51	0.79	1.47	1.11	2.52	3.23	4.29
12	0.38	0.52	0.76	1.31	1.64	2.92	3.27	4.31
13	1.38	2.13	3.74	4.95	7.34	11.37	14.68	20.17
14	0.54	0.74	1.19	2.35	2.25	3.76	4.28	8.01
15	0.94	0.98	1.63	2.30	3.82	4.24	5.78	9.35
16	0.31	0.32	0.65	1.24	1.30	1.82	2.21	3.72

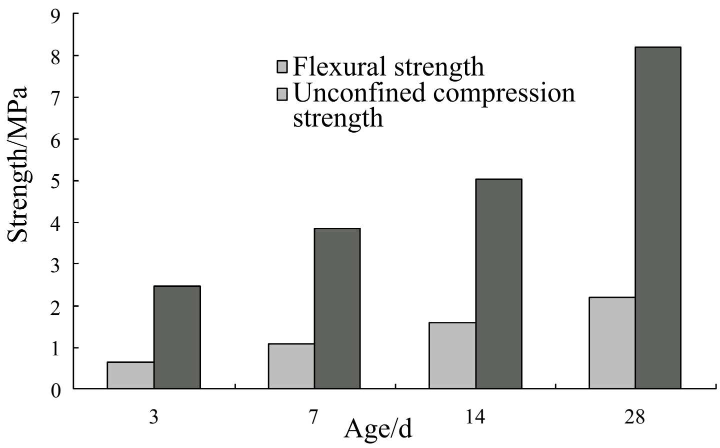

Fig. (4). Histogram of bending and compressive strength increase with time.

As can be seen from Table **2**, the composite slurry concretion with a water-solid ratio of 0.5**:**1, showed the largest bending strength and unconfined compressive strength. At a water-solid ratio of 0.8**:**1, the mechanical properties of the 3rd and 7th groups were better. At a water-solid ratio of 1:1, the mechanical properties of the 10th and 14th groups were better, but the strength of the slurry concretion with this water-solid ratio was generally low.

Taking account of the lower cost of cement slurry with a high level of fly ash, Group 3 (water-solid ratio of 0.8:1) was selected as the optimized composite slurry.

3. MAXING TEST OF CEMENT-SODIUM SILICATE SLURRY

3.1. Experiment Design

A P.O.32.5R cement-sodium silicate (CS) mix with a modulus of 3.0 was used in the experiments. Three factors affecting the basic properties such as the CS slurry setting time, the concretion strength, and the concretion rate were the water-cement ratio, the sodium silicate concentration, and the CS volume ratio.

3.2. Experimental Results for Setting Time

3.2.1. The Influence of the Water-cement Ratio on the Setting Time

This group of experiments was conducted using a fixed sodium silicate concentration of 36Be' and a fixed CS volume ratio of 1:1. The results are shown in Fig. (**5**).

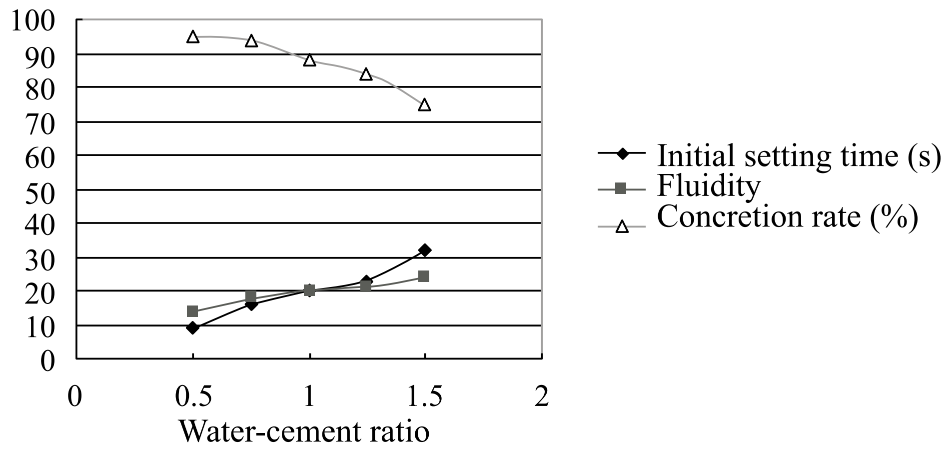

Fig. (5). Relation curve of water-cement ratio with setting time.

Generally, a lower water-cement ratio was associated with a faster reaction time between the cement and sodium silicate, and a shorter setting time.

3.2.2. The Influence of Sodium Silicate Concentration on the Setting Time

This group of experiments was carried out using a fixed water-cement ratio of 1:1 and a fixed CS volume ratio of 1:1. The results are shown in Fig. (**6**).

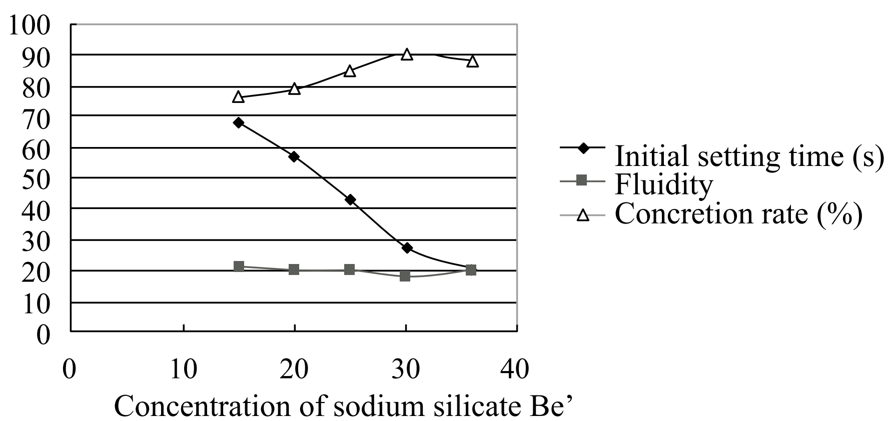

Fig. (6). Relation curve of sodium silicate concentration with setting time.

As can be seen from Fig. (**6**), the initial setting time of the cement-sodium silicate varied significantly with the concentration (15~36 Be'). If the initial setting time is excessive when grouting in flowing water, the gel properties will change greatly according to the flow velocity of water, the flow pressure of water, and the contact area of slurry and water. If the slurry supply is insufficient, the slurry will usually not set. Note: ① If the sodium silicate concentration is too low, bonding of the cement cannot be achieved; ② The setting time of the cement increases as the sodium silicate concentration is reduced.

3.2.3. The CS Volume Ratio Influences on the Setting Time

This group of experiments was carried out using a fixed water-cement ratio of 1:1 and a fixed sodium silicate concentration of 36Be'. The experimental results are shown in Fig. (**7**).

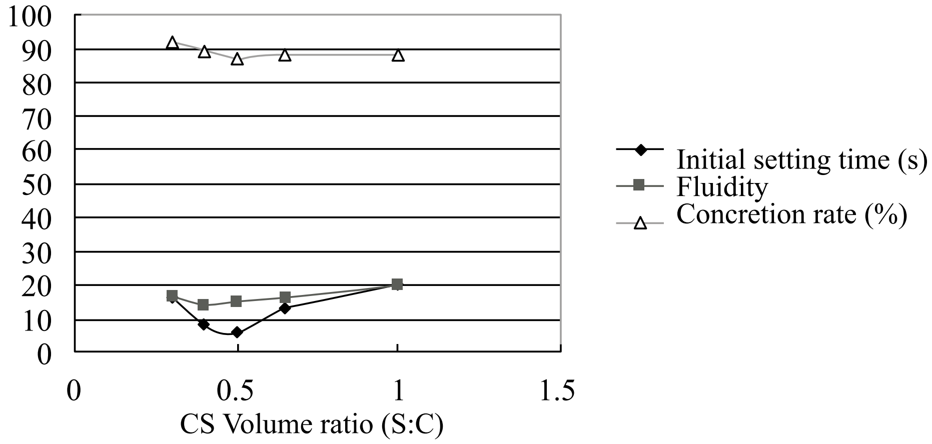

Fig. (7). Relation curve of CS volume ratio with setting time.

As can be seen from Fig. (**7**), the cement slurry with 50% sodium silicate by volume had the shortest initial setting time.

3.3. Experimental Results of Concretion Strength

The compressive strength of the CS slurry initially increased quickly, reaching 70%~90% of its final strength within about 7d. This is favorable for real grouting applications. The following factors affect the compressive strength of CS slurry:

3.3.1. The Water-cement Ratio

This group of experiments was carried out using a fixed sodium silicate slurry concentration of 36Be' and a fixed CS volume ratio of 1:1, as shown in Fig. (**8**).

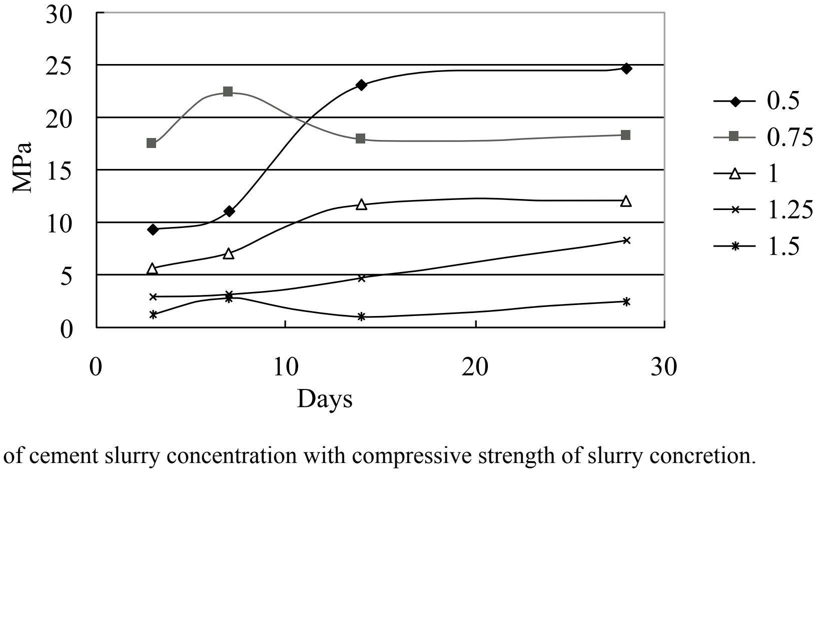

Fig. (8). Relation curve of cement slurry concentration with compressive strength of slurry concretion.

As can be seen from Fig. (**8**), a higher cement concentration can increase the later compressive strength.

3.3.2. The Influence of Sodium Silicate Concentration.

This group of experiments was carried out using a fixed water-cement ratio of 1:1 and a fixed CS volume ratio of 1:1, as shown in Fig. (**9**).

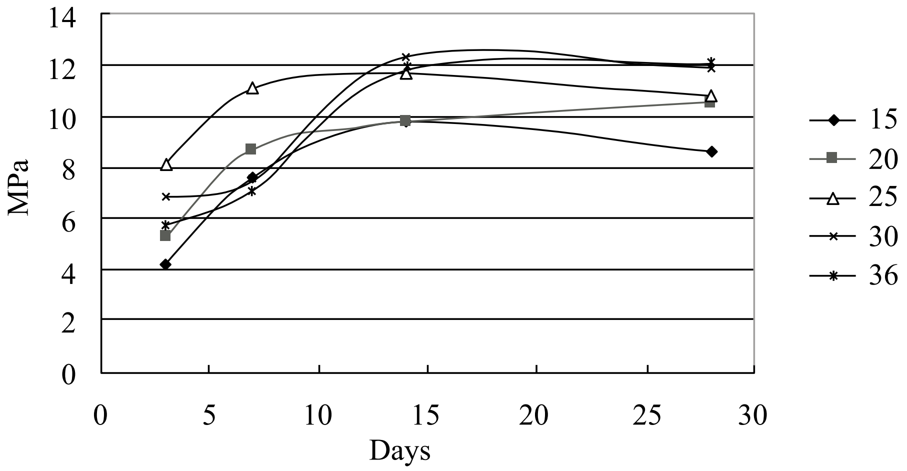

Fig. (9). Relation curve of sodium silicate concentration with compressive strength of slurry concretion.

Many studies have investigated the relationship between sodium silicate concentration and the compressive strength of CS slurry, and the following laws have been obtained: ① The change in compressive strength of CS takes the form of a curve, with a peak strength at about day 14. This is because precipitation of NaOH during setting adversely affects the later strength of the concretion. ② The early strength of the concretion increases fastest at a sodium silicate concentration of 25Be'. This means that an appropriate matching ratio exists in the reaction between cement and sodium silicate, at which the reaction completes and the strength is maximized.

3.3.3. Influence of CS Volume Ratio

This group of experiments was carried out using a fixed water-cement slurry ratio of 1:1 and a fixed sodium silicate concentration of 36Be', as shown in Fig. (**10**).

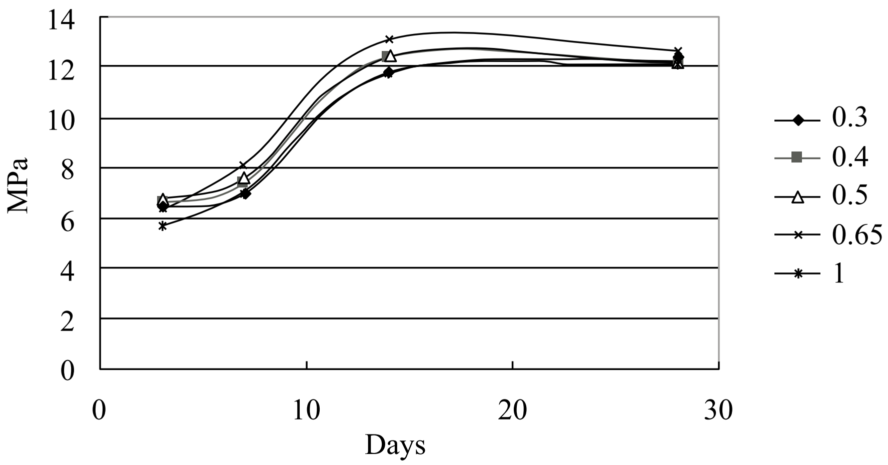

Fig. (10). Relation curve of CS volume ratio with compressive strength of slurry concretion.

It can be seen that, at a cement slurry ratio of 1:1, the relation between compression strength and sodium silicate concentration was complex. This reflects the complexity of the chemical reaction between cement and sodium silicate, and is independent of the C: S volume ratio within a certain range.

4. GROUTING STRENGTHENING TEST COMPARISON AND ENGINEERING APPLICATION

To compare the strengthening effect of the optimized composite slurry and cement-sodium silicate, two kinds of slurry were prepared. The first was a cement-sodium silicate slurry in which the ratio of water to cement was 0.8:1, the density of sodium silicate was 36Be', and the volume ratio of cement to sodium silicate was 1:1. The second was the optimized composite slurry (Group 3).

A mould 100mm × 100mm × 100mm was used. A pulverized coal sample from the No. 6 Drill Site of the 3^{-1} Coal Seam of the Ningtiaota Coal Mine was used. A sample with a bulk density of 1.28 × 10^3kN/m^3 and grain diameter of 5 mm<<20mm was placed in the mould Figs. (**11a** and **11b**) show the coal sample before and after strengthening). The formulated slurry was poured into the mould, fully stirred and compacted, and demoulded after curing. The non-lateral confined compressive strength at 20 ± 5°C and humidity >90% was recorded at different times, as shown in Table **3**.

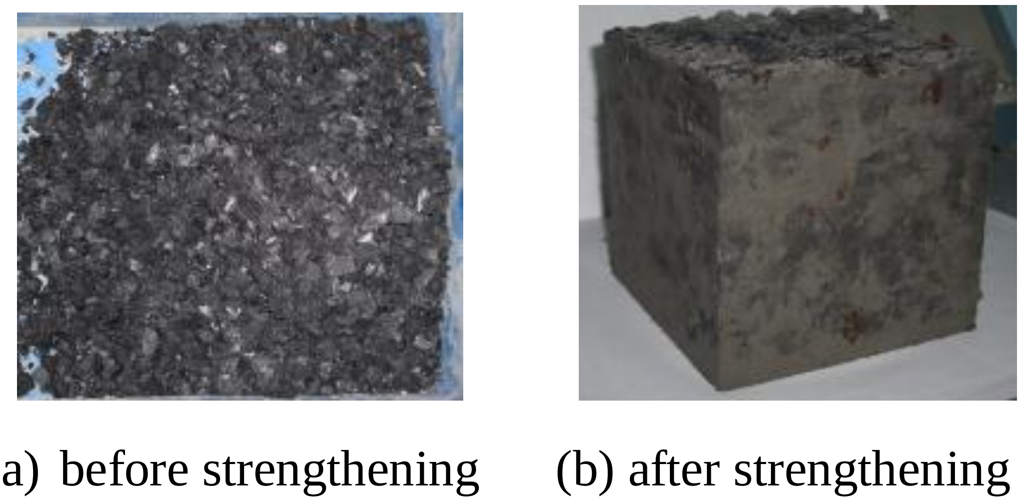

Fig. (11). Broken coal sample.

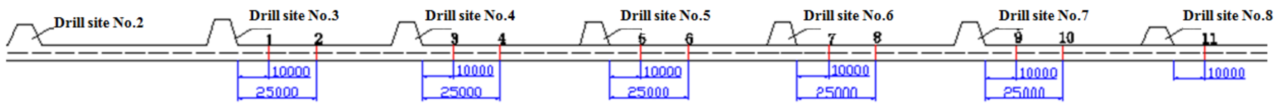

Fig. (12). Arrangement for displacements monitoring points at both sides of the roadway.

Table 3. Non-lateral confined compressive strength.

Grouting type	Non-lateral compressive strength (MPa)			
	3(d)	7(d)	14(d)	28(d)
Cement-sodium silicate slurry (0.8:1)	5.325	6.760	10.614	8.301
Optimized composite slurry (Group 3)	1.814	2.097	2.851	5.085

The results confirmed that the optimized composite slurry had a lower non-lateral confined compressive strength than the cement-sodium silicate slurry with the same water-solid ratio. However, the addition of large amounts of fly ash to the optimized composite slurry significantly reduced the cost, while the concentration of H_2S decreased effectively. Therefore, in a water-borne H_2S environment, the optimized composite slurry improved both the grouting and the strengthening results effectively.

Based on a large number of in-situ experiments, the selection of slurry for grouting in Water-borne H_2S environment of coal or rock mass must take account of the water level (high, medium, low) at the grouting point and the H_2S concentration; application scope for the two kinds of slurry is shown in Table **4**.

Table 4. Application scope for the two kinds of slurry.

Slurry application scope	H_2S gas concentration (ppm)	Water outlet ($m^3 \cdot min^{-1}$)	Proportion			Initial setting time(s)	Concretion rate (%)	Compressive strength (MPa)			
			Water-cement ratio	Sodium silicate concentration (Be')	CS volume ratio			3(d)	7(d)	14(d)	28(d)
Cement-sodium silicate	0~0.05	10~30	0.75	36	0.4	7	96	16.9	21.8	18.2	19.4
		1~10	1	36	0.35	10	91	6.7	7.2	11.9	12.1
		0~1	1.2	36	0.3	24	84	3.2	4.3	5.6	8.7
Optimized composite slurry	0.05~50	0~10	Water-solid Ratio 0.8:1			13:15 (h/min)	93	2.48	3.85	5.03	8.18

The *in situ* experiments have shown that the gel property of CS double slurry changes greatly, depending on the flow velocity of the water at the point to be plugged, the water outlet, and the contact area of slurry and water. If insufficient slurry is supplied, it is usually not set. Our experiments suggest the following: the CS double slurry is unlikely to set when the water outlet is larger than $1m^3/h$ and a single outlet section is larger than $30m^3 \cdot min^{-1}$. The water flow must therefore be limited using a slipping chuck, saw dust, textiles, rubber sheeting, or other means in order to allow viscous flow of the slurry. For successful grouting, the slurry supply should generally be no lower than 6 m^3/h. If the H_2S gas concentration is too high, it must be reduced using methods such as sprinkling with quicklime. To guarantee the safety of personnel on site, the optimized composite slurry should be used to eliminate the hidden danger presented by H_2S gas.

At present, tests are being conducted on the grouting and strengthening of the central auxiliary transport roadway of the 3^{-1} coal seam of the Ningtiaota Coal Mine operated by the Shaanxi Coal Industry Group. On-site tests have been conducted using the optimized composite slurry, with ratio of cement: fly ash: clay: quicklime: sodium sulfate: water as 1:0.45:0.20:0.05:0.07:1.32.

At the Ningtiaota Coal Field, atmospheric precipitation supplies a flow to the 3^{-1} coal seam from southeast to northwest. Parts of the 3^{-1} coal seam are rich in groundwater and contain H_2S.This threatens future shaft construction and mining. Following a geological engineering overview, it was decided to use a grouting method to reinforce the 3^{-1} coal auxiliary transport roadway, to allow its safer long-term use. At the same time, this can reduce residual water seepage at the coal wall at the side of the roadway. Based on the water level and H_2S concentration, seven drill sites were selected (Fig. **12**).

Because new optimized composite slurry was used, testing took place only at drill site No. 6 (a crushed coal seam), while a cement-sodium silicate slurry was used at the other drill sites. Since the water flow in the grouting borehole was complex, the CS double fluid was changed to match the water outlet. The *in situ* experiments suggested that the method was able to completely plug the water ingress within 30 minutes. After 20 days of grouting, almost no water seepage was observed at the two sides. RAMAC geological radar was used to investigate the left coal wall, and the detection images confirmed a relatively uniform grouting curtain. This confirmed that the optimized composite slurry had achieved its intended goal.

CONCLUSION

This paper presented an introductory study on the properties of composite slurry and cement-sodium silicate slurry; the results are as follows:

1. Orthogonal testing was used to analyze the physical and mechanical properties of composite slurry of different proportions. The slurry with a water-solid ratio of 0.8:1 was shown to have the optimal combination of properties. The addition of large amounts of fly ash can reduce the cost of the slurry. The 3^{rd} group of slurry (mass ratio of cement: fly ash: clay: quicklime: sodium sulfate: water is 1:0.45:0.20:0.05:0.07:1.32) was found to be the optimal composite slurry. Range analysis results suggested that the water-solid ratio is the key factor determining the properties of the slurry. Sodium silicate was then added to the optimized composite slurry (Baume degree of sodium silicate 35, ratio by volume of sodium and cement 0.6:1). The initial setting time of the slurry was found to be 10s and little impact was found on later strength, suggesting that sodium silicate can

be used effectively to adjust the setting time of composite slurry and make the slurry controllable.

2. A uniform experimental method was used to investigate the impact of the water-cement ratio, concentration of sodium silicate, and volume of CS on the setting time, consolidation strength, and concretion rate of the CS slurry.
3. This paper provides the application range of two grouting materials (optimized composite slurry and CS slurry), based on a synthesis of experimental data and a large number of *in situ* trials.
4. Curing tests were performed on a smashed coal sheet using optimized composite slurry and a cement sodium silicate slurry (water-cement ration of 0.8:1, density of sodium silicate 36Be', ratio by volume of cement to sodium silicate 1:1). The non-lateral confined compressive strength of consolidation at different times was measured. The results demonstrated that the optimized composite slurry had a lower non-lateral confined compressive strength than the cement sodium silicate slurry with the same water-solid ratio. However, the addition of large amounts of fly ash to the optimized composite slurry significantly reduced its cost, while the concentration of H_2S decreased effectively. Therefore, in a water-borne H_2S environment, the optimized composite slurry was shown to improve both grouting and strengthening. From the combined data, the scope of application of the two types of grouting materials was derived.
5. The optimized composite slurry and CS slurry were applied to the grouting reinforcement of the center auxiliary transport roadway of the 3^{-1} coal seam at the Ningtiaota Coal Mine, operated by the Shaanxi Coal Industry Group. The type and formula of the slurry could be changed in a timely manner, based on the water outlet and concentration of H_2S at the site, achieving successful grouting reinforcement and seepage-proofing.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflict of interest.

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