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Effect of Nano-$CaCO_3$ on the Sealing Efficiency of Grouts in Flowing Water Grouting

Yao-Hui Liu[a,b], Ping Yang[a,c], Ze-Bin Ouyang[a], Xiao Wei[b], and Lisha Zhang[d]

[a]Dept. of Geotechnical Engineering, College of Civil Engineering, Tongji University, Shanghai 200092, China
[b]Dept. of Civil and Environmental Engineering, National University of Singapore, Singapore 119077, Singapore
[c]Key Laboratory of Geotechnical and Underground Engineering of Ministry of Education, Tongji University, Shanghai 200092,China
[d]Dept. of Civil Engineering, Zhejiang University City College, Hangzhou 310015, China

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ABSTRACT

Flowing water grouting is a big challenge in the tunneling and underground engineering. To enhance the early strength and grouting effectiveness of slurry for flowing water grouting, nano-$CaCO_3$ and fly ash were mixed with cement based grout. A series of physical simulation tests were conducted to simulate the flowing water grouting process in rough rock fracture, and investigate the effect of nano-$CaCO_3$ content on the fluid pressure and sealing efficiency of grouts. Results of viscosity tests show that the viscosity of grouts decreased with an increase of nano-$CaCO_3$ content. Scanning electron microscope (SEM) tests indicate that nano-$CaCO_3$ can promote the formation of fibrous hydrates and enhance the flowing water resistance of grouts. Increasing nano-$CaCO_3$ content resulted in the first increase while later decrease of maximal fluid pressure (MFP) and sealing efficiency (SE) of grouts. Reducing water cement ratio of grouts and incorporating fly ash can effectively improve the SE of flowing water grouting.

1. Introduction

Grouting is an effective method to prevent groundwater inrush and enhance the rock mass in tunneling engineering (Li et al., 2016; Zhang and Sun, 2019). However, the solidifying rate and early strength of traditional cemented based grouts can't meet the requirement for flowing water grouting. Therefore, studies on the improvement of characteristics and grouting effectiveness of grouts in flowing conditions draw more attention in recent years.

Cement based grouts and chemical grouts are most widely used in flowing water grouting (Zhou et al., 2019; Liu et al., 2020). However, the cost of chemical grouts is high and chemical grouts may pose environmental and health hazards (Zhang et al., 2019). Different from the cement used in construction and building, early strength is especially important for cement applied in flowing water grouting. Flowing water will rinse out low early strength grouts, resulting in the decrease of grouts concentration and the failure of grouting (Yang et al., 2018). Therefore, it's an urgent need to acquire a kind of environment-friendly grouts with low cost and high early strength for flowing water grouting.

The application of nanomaterials in cement was widely studied due to their large specific surface area and exceptional properties (Muhd Norhasri et al., 2017; Jiang et al., 2018; Behzadian and Shahrajabian, 2019; Ehsani et al., 2017; Irshidat and Al-Saleh, 2018). Nano-silica was proven to densify and refine the micro voids of cement (Lindgreen et al., 2008; Rostami et al., 2019). Nano-alumina can act as dispersion agent and speed up the initial setting time as mixing with cement (Jo et al., 2007; Nazari and Riashi, 2011). Carbon nano tube can improve the flexibility and enhance the strength of cement (Morsy et al., 2011; Rostamiyan et al., 2015). However, the application of nanomaterials in actual engineering was rare due to the high cost of nanomaterials. Therefore, nano-$CaCO_3$ with low cost was applied to improve characters of cement in recent years (Shaikh and Supit, 2015; Sato and Beaudoin, 2011). Nano-$CaCO_3$ has been proved to have an influence on the microstructure (Shaikh et al., 2017), flowability (Wang et al., 2016), durability properties (Supit and Shaikh, 2014), water demand (Qian et al., 2008), hydration

CORRESPONDENCE Ping Yang pingyang@tongji.edu.cn Dept. of Geotechnical Engineering, College of Civil Engineering, Tongji University, Shanghai 200092, China; Key Laboratory of Geotechnical and Underground Engineering of Ministry of Education, Tongji University, Shanghai 200092,China

(Sato and Beaudoin, 2011) and compressive (Shaikh and Supit, 2014) of cement. Camiletti et al. (2013) suggested that nano-$CaCO_3$ can accelerate the setting and hardening process of concrete through providing nucleation sites. Nano-$CaCO_3$ was also conducive to increase the early strength of concrete (Meng et al., 2017). However, more attentions are concentrated on the application of $CaCO_3$ in the cement used in the construction, and the investigation on the application and influence of nano-$CaCO_3$ in flowing water grouting is rare. Therefore, nano-$CaCO_3$ was incorporated with cement based grouts to improve the early strength and grouting effectiveness of grouts in flowing water condition in this work.

The overarching objective of this work was to determine effects of nano-$CaCO_3$ on the characters and grouting effectiveness of nano-$CaCO_3$ based grouts in rough fracture and flowing water condition. The fracture surface was usually simplified as smooth in previous studies, which will decrease the accuracy of grouting (Sui et al., 2015; Li et al., 2016). Therefore, a fracture simulation device with rough fracture rather than smooth surface was created and used to simulate flowing water grouting process of nano-$CaCO_3$ based grouts. The specific objectives were as follows: 1) determine effects of nano-$CaCO_3$ on the viscosity of grouts; 2) understand the microscopic mechanism of the influence of nano-$CaCO_3$ and fly ash; 3) investigate effects of nano-$CaCO_3$ on sealing efficiency (SE) and fluid pressure of grouts through flowing water grouting simulation test; 4) verify the possibility of improving SE of nano-$CaCO_3$ composited grouts through reducing water cement ratio (W/C) and incorporating fly ash.

2. Materials and Methods

2.1 Materials

P.O 42.5 cement was provided by Hailuo Cement Company. Nano-$CaCO_3$ with 40 – 80 nm diameter was produced by Xianfeng company and the physical properties of nano-$CaCO_3$ are

Table 1. Physical Properties of $CaCO_3$

Form	Diameter (nm)	Bulk density (g/ml)	PH
White power	40 – 80	0.40	9.50 – 10.0

Table 2. Chemical Composition of OPC, Fly Ash and Nano - $CaCO_3$

	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO
OPC	23.12	5.73	3.69	64.84	2.75
Fly ash	53.62	24.16	8.16	1.45	2.04
Nano-$CaCO_3$	-	-	0.01	98	0.4

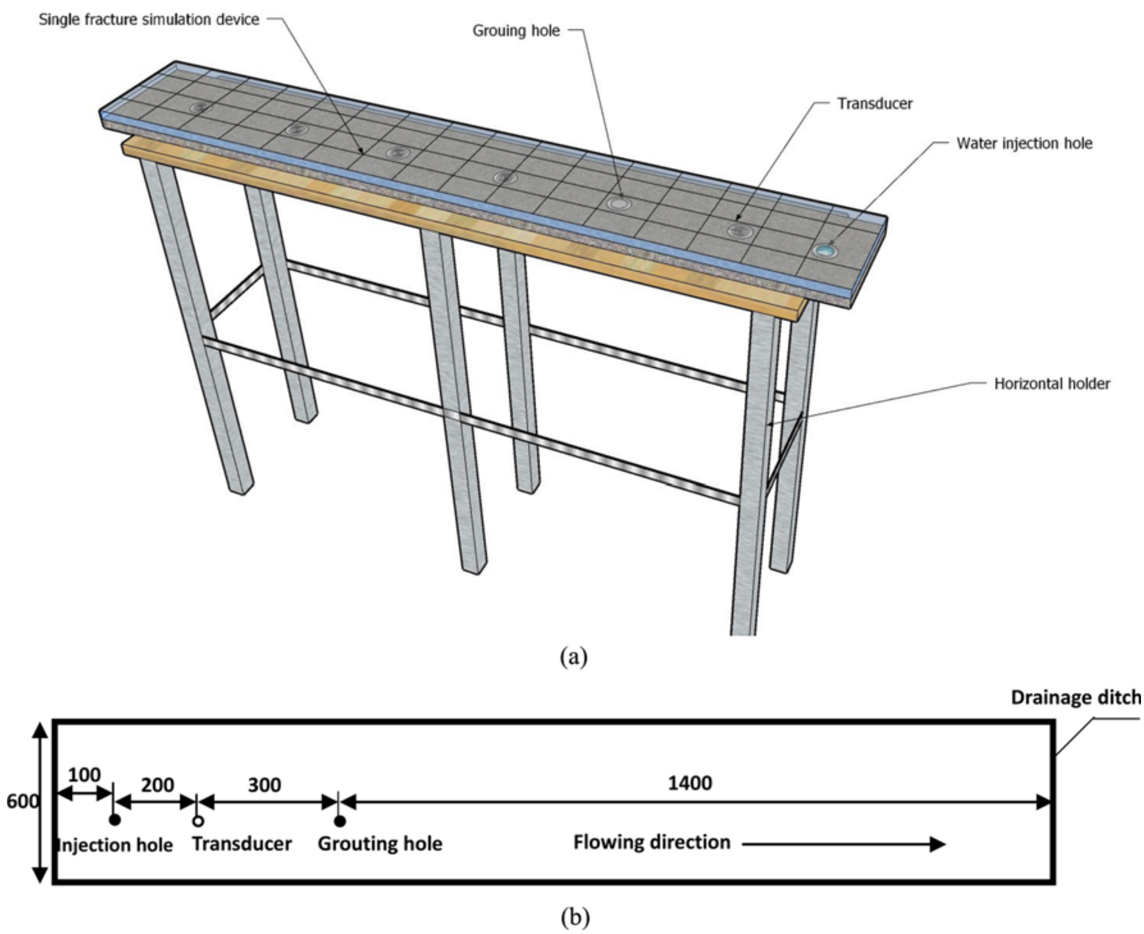


Fig. 1. Flowing Water Grouting Simulation Device: (a) Scheme Diagram, (b) Plan View (unit: mm)

summarized in Table 1. Fly ash with grade II was provided by Huifeng Company. The chemical components of cement, nano-$CaCO_3$ and nano-$CaCO_3$ are summarized in Table 2.

2.2 Experimental Set-Up

Process of flowing water grouting was simulated by a self-designed set-up, as shown in Fig. 1. The simulation device comprise horizontal holder, rough fracture simulation device and grouting system (Liu et al., 2020). Roughness of fracture surface has been proven to have an influence on the flowing water grouting (Yang et al., 2018; Scesi and Garrinoni, 2007; Jin et al., 2019).The rough fracture simulation device could be used to simulate the rough rock fracture surface in practical engineering. Different from the smooth fracture surfaces in previous study (Sui et al., 2015; Liang et al., 2019; Zou et al., 2020), the fracture surface in experiment is uneven and designed on the basis of Barton Curve. Barton and Choubey (1977) proposed Barton curves on basis of the fluctuation of hundreds of fracture surface in practical engineering and used these curves to describe the roughness of rock fracture surface, as shown in Fig. 2. The manufacture method and feature of rough fracture surface have been introduced in details in author's previous literatures (Yang et al., 2020). The roughness of fracture used in the experiments is 12 – 14. The equivalent hydraulic width is 7.3 mm and the concept of equivalent hydraulic width has been explained in details in author's previous study (Yang et al., 2018).

The physical parameters of grouting machine and water pump

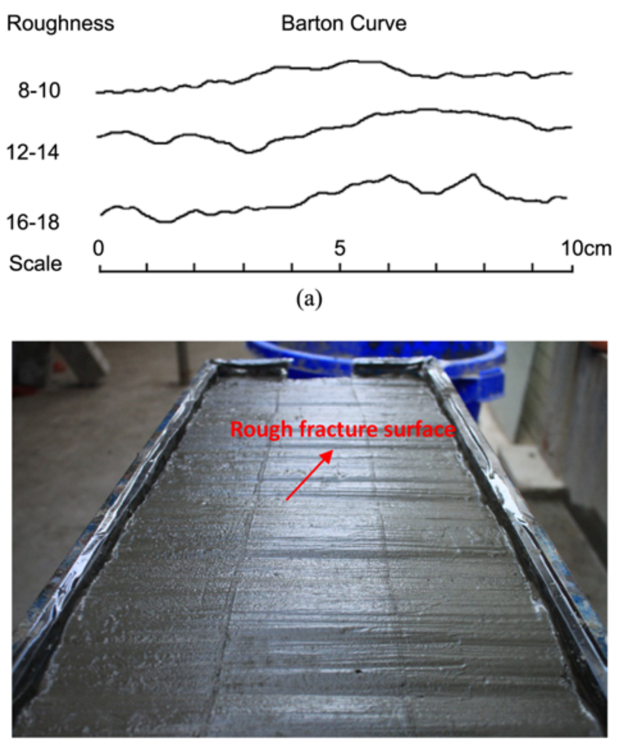


Fig. 2. Rough Fracture Simulation Device: (a) Three Typical Barton Curve, (b) Rough Fracture Surface

Table 3. Component of Grouts in Viscosity Test

Number	Fly ash	Nano-$CaCO_3$	W/C
1	0	0%	0.8
2	0	0.5%	0.8
3	0	1%	0.8
4	0	1.5%	0.8
5	0	2%	0.8

have also been introduced in author's previous literature (Yang et al., 2018; Yang et al., 2020). Water was injected into fracture with constant pressure which agreed with flowing water grouting in practical engineering.

2.3 Viscosity Test

Viscosity test was carried out to inspect effects of nano-$CaCO_3$ on the viscosity of grouts. Components of grout in each viscosity test are introduced in Table 3. The additive comprised of 60% sodium aluminate, 20% sodium silicate, 14% sodium chloride and 6% Hydroxypropyl methyl cellulose (HPMC) (Yang et al., 2020). The mass percent of additive in grout is 6%. The component and mass percent of additive has been proved to be optimal to adjust the solidification rate of grout (Yang et al., 2018).

The volume of grout in each viscosity test is 450 ml. Additive and nano-$CaCO_3$ was putted into a beaker. Then water was poured into the beaker and stirred uniformly. Then cement was poured into beaker and mixed with 200 r/s for 50s. Finally the viscosity of grout was measured using a viscometer (NSJ-9S, China) at an interval of 2 minutes. The W/C of viscosity test (0.8) was different from that in other tests (0.7) because the viscosity of cement with 0.7 W/C exceeds the measuring range of viscometer. However, the effects of nano-$CaCO_3$ on the viscosity of grouts with 0.7 W/C and 0.8 W/C are similar.

2.4 Flowing Water Grouting Simulation Test

2.4.1 Sealing Efficiency

Flowing water grouting tests were carried out to investigate the

Table 4. Grout Components in Flowing Water Grouting Test

Number	Fly ash	Nano-$CaCO_3$	W/C
1	0	0%	0.7
2	0	0.5%	0.7
3	0	1%	0.7
4	0	1.5%	0.7
5	0	2%	0.7
6	40%	0%	0.6
7	40%	0.5%	0.6
8	40%	1%	0.6
9	40%	1.5%	0.6
10	40%	2%	0.6

SE and maximal fluid pressure (MFP) of grouts with various components. SE is a significant index to assess the grouting effectiveness of grouts in fracture (Sui et al., 2015). Flowing water with 250 ml/s and 20 kPa was used in grouting simulation tests. Then fly ash, nano-$CaCO_3$ and cement was mixed with water as Table 4 and stirred for 50s. After 40s standing, slurry was supplied with a grouting machine. The grouting rate was 60 ml/s and grouting time is 5 min. Flowing rate after grouting in each test was recorded. In test 1 – 5 (Table 4), effects of nano-$CaCO_3$ on SE and MFP were investigated. In test 6 – 10, the W/C was reduced to enhance the strength of grout and 40% fly ash was used to increase the fluidity of slurry.

2.4.2 Maximal Fluid Pressure

The solidification of slurry resulted in the decrease of flowing rate and a rapid increase of hydraulic pressure before grouting hole during grouting process. The pressure caused by flowing water is named as fluid pressure and the maximal pressure that grouts can endure is the MFP. The slurry will be rushed out from fracture when the fluid pressure exceeds the MFP of solidified grouts (Sui et al., 2015). Therefore, MFP has a crucial influence on the effectiveness of flowing water grouting, especially when the hydraulic pressure is great. The maximal compressive strength (1d and 3d) of nano-materials composited cement is widely investigated in previous studies (Supit et al., 2014). However, the maximal compressive strength can only reflect the strength of completely solidifying cement, which couldn't reflect the manifestation of slurry in flowing water grouting. Therefore, MFP rather than maximal compressive strength of grout was investigated in this work.

The measuring method of MFP was similar to that of SE. Water was injected into fracture with 250 ml/s. But the water pressure was 30 kPa which was different from SE test. The component of slurry agreed with SE test. The grouting rate was 60 ml/s and Grouting time was 2 min. Finally, fluid pressure during grouting process was determined by a sensor before the grouting hole.

2.5 Scanning Electron Microscope Tests

Scanning electron microscope (SEM) tests were carried out to understand effects of nano-$CaCO_3$ and fly ash on grouts in microscopic perspectives (Lilkov et al., 2011). The slurry samples in SEM tests consist of samples without nano-$CaCO_3$ and fly ash, samples with 2% nano-$CaCO_3$, and samples with 40% fly ash and 2% nano-$CaCO_3$. Size of samples was 14 mm × 6 mm × 5 mm. The samples were dried using liquid nitrogen and scanned using SEM.

3. Results and Discussion

3.1 Results of Viscosity Test

The development of viscosity of grouts with various nano-$CaCO_3$ is presented in Fig. 3. Viscosity of grouts increase with time (Fig. 3), and the increasing velocity is much greater than traditional nano-$CaCO_3$ based cement due to the acceleration effect of additive (Zhang et al., 2019). Rapid solidification of nano-$CaCO_3$ composited grouts ensured that slurry can solidify in time before being washed out and improved the grouting effectiveness in flowing water condition.

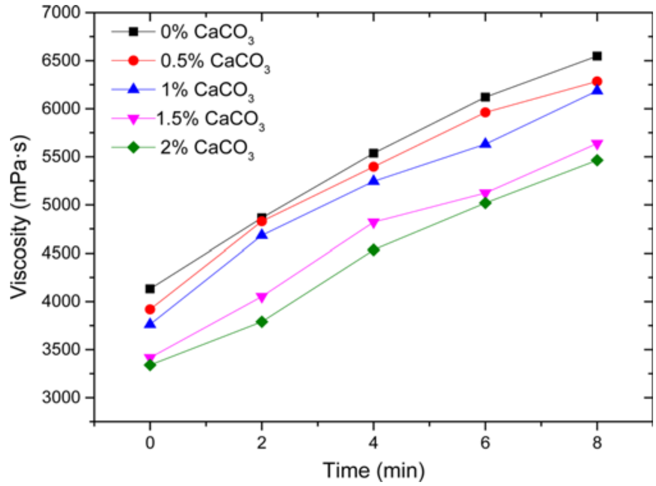


Fig. 3. Variations in Viscosity of Grouts with Various Nano-$CaCO_3$ Contents

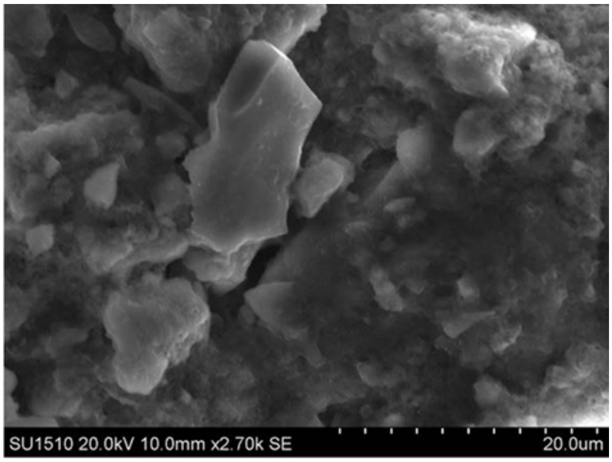
There is no uniform conclusion on the effect of nano-$CaCO_3$ on the viscosity of cement. The viscosity increased with nanomaterial content because the larger specific surface area and more surface water in some studies (Kong et al., 2013; Quercia et al., 2012). Zhang and Sun (2019) found that the increasing nanomaterial will reduce viscosity when the nanomaterial content is smaller than 0.5%. However, increasing nanomaterial can result in the raise of viscosity when nanomaterial content exceeded 0.5%.

Figure 3 shows that the viscosity of grouts with 2% nano-$CaCO_3$ was always smallest. Increasing nano-$CaCO_3$ content was conducive to the decrease of grouts viscosity. However, the increasing rate was similar among grouts with various nano-$CaCO_3$ contents. Nano-$CaCO_3$ enlarged the density of cement particles and decrease the gap between cement particles, resulting in an increase of free water and fluidity of grouts. Additionally, nano-$CaCO_3$ disturbed the physical contacting and chemical reaction between additive and cement, resulting in the decrease of accelerating effect of additive. All these reasons resulted in the decrease of slurry viscosity after incorporating nano-$CaCO_3$.

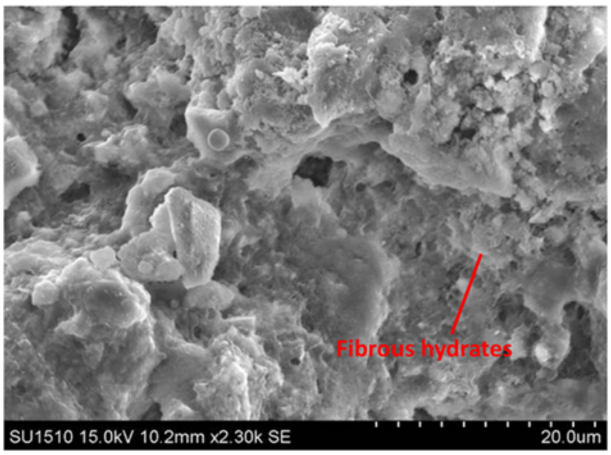
3.2 Results of SEM Test

The SEM images of grout samples are shown in Fig. 4. Figs. 4(a) and 4(b) show that more fibrous hydrates were formed when 2% nano-$CaCO_3$ were added into grout, indicating that nano-$CaCO_3$ can promote the hydration of grout. Flowing water resistance is an important characteristics of grout used for describing the stability of grouting in flowing water condition (Yang et al., 2018). Grouts with higher flowing water resistance can keep better stability and higher concentration in flowing water condition. Grouts with lower flowing water resistance had a higher possibility to be scatter and washed out by flowing water. The fibrous hydrates connected with other hydration products and enhanced the cohesion force of hydration products, resulting in the increase of flowing water resistance of grouts, which was conductive to the improvement of grouting effectiveness.

There were a great amount of spherical fly ash particle in grout with 40% fly ash (Fig. 4(c)). Therefore, fly ash can increase the fluidity of grouts due to the 'ball effect', which agrees with previous studies (Meng et al., 2017).



(a)



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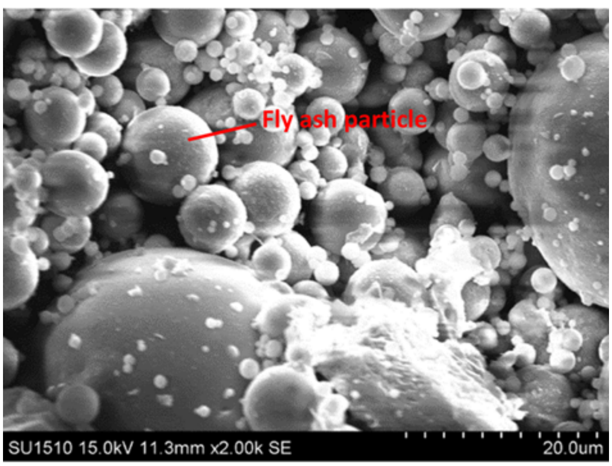
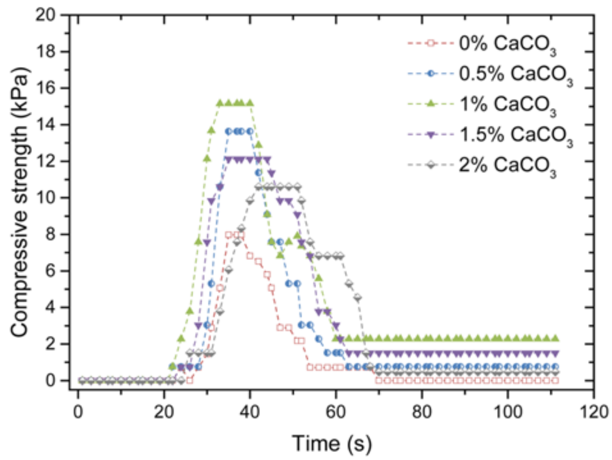


(c)

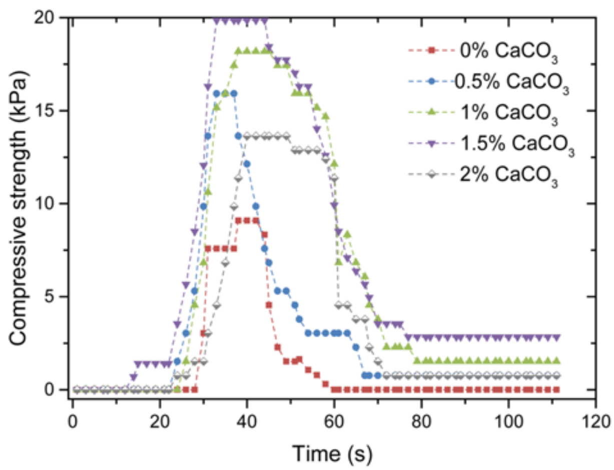
Fig. 4. SEM Image of Grouts: (a) Without Nano-$CaCO_3$, (b) With 2% $CaCO_3$, (c) With 2% $CaCO_3$ and 40% Fly Ash

3.3 Development of Fluid Pressure

For the readability of this work, the development of grouts with 0.7 W/C (Test 1 – 5) and grouts with 0.6 W/C and 40% fly ash (Test 6 – 10) are shown in Figs. 5(a) and 5(b), respectively. The MFP of grouts without nano-$CaCO_3$ was smaller than that of grouts with nano-$CaCO_3$ (Fig. 5). The MFP of grouts with 1% nano-$CaCO_3$ content was greatest (15.15 kPa) when the W/C was 0.7 (Fig. 5(a)). This indicates that MFP enlarged with nano-$CaCO_3$ content in initial stage. Then increasing nano-CaCO3 reduced MFP when nano-$CaCO_3$ surpassed a certain value. Nano-$CaCO_3$ can enhance the early strength of grouts due to the nucleation of hydration products and the nano-filler effects induced by the incorporation of nano-$CaCO_3$ (Camiletti et al., 2013). Additionally, existence of nano-$CaCO_3$ has been proven



(a)



(b)

Fig. 5. Development of Fluid Pressure of Grouts with: (a) 0.7 W/C, (b) 0.6 W/C and 40% Fly Ash

to improve the flowing water resistance of grouts (In Section 3.2). Therefore, the MFP increased with nano-$CaCO_3$ content in the initial stage. However, increasing nano-$CaCO_3$ reduced the viscosity of grouts, resulting in grouts being washed out by the flowing water as the nano-$CaCO_3$ surpassed a certain value. Therefore, there should be a limit to the nano-$CaCO_3$ content for ensuring MFP of grouts when hydraulic pressure is high.

The duration time of MFP increased with nano-$CaCO_3$ content (Fig. 5). For instance, the duration time increased from 3 s to 6 s when nano-$CaCO_3$ content raised from 0% to 2% (Fig. 5(a)). It's worth noting that increasing nano-$CaCO_3$ results in longer duration time when nano-$CaCO_3$ exceeded 1% and the MFP started to decrease. The nano-$CaCO_3$ improved the microstructure of grouts and enhanced early strength, resulting in that grouts became more stable and can suffer high fluid pressure for longer time when nano-$CaCO_3$ content incerased.

Although nano-$CaCO_3$ can improve the MFP of grout, the improvement was limited. Therefore, W/C was decreased to enhance the fluid pressure and strength of grouts. However, experimental results indicates that the flowability of grouts with 0.6 W/C and various $CaCO_3$ content was terrible, resulting in the blocking of grouting machine and failure of grouting. Fly ash was a widely used materials to enhance the flowability and strength of grout due to its environmentally friendliness, low cost and high effectiveness. Therefore, fly ash (40%) were added into the grouts with 0.6 W/C to increase the flowability and pumpability. Fig. 5(b) suggests that the MFP of grouts with 0.6 W/C and 40% fly ash was much greater than that of grouts with 0.7 W/C. For instance, the range of MFP of grouts with 0.6 W/C was 9.09 kPa – 19.86 kPa, while that of grouts with 0.7 W/C was 7.97 kPa – 15.15 kPa.

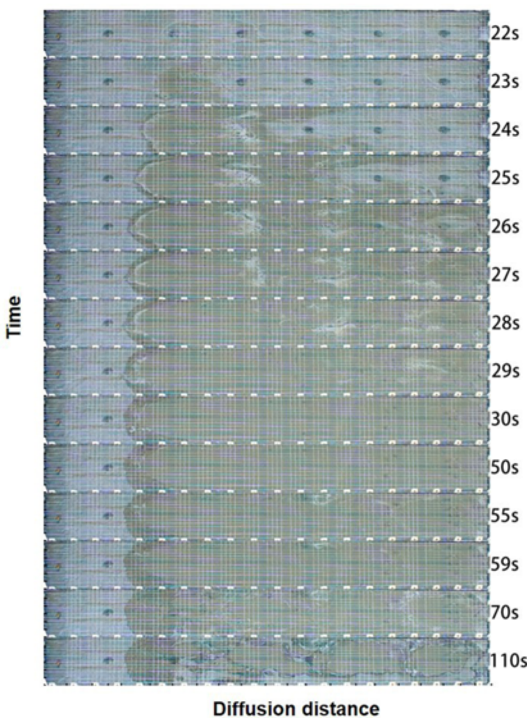


Fig. 6. Diffusion Process of Slurry in Grouting Process in Test 3

Influence of nano-$CaCO_3$ on the MFP of grouts with 0.6 W/C and 40% fly ash was similar to that of grouts with 0.7 W/C. Fig. 5 indicates that 0.6 W/C, 40% fly ash and 1.5% nano-$CaCO_3$ content was the optimal composition of grouts to enhance its MFP. For the convenient understanding of grouting process, diffusion process of slurry in Test 3 (1% $CaCO_3$ and 0.7 W/C) is presented in Fig. 6. As shown in Fig. 6, the time when grouts began to solidify (about 24 s) agreed with the time when fluid pressure started to increase (Fig. 5).

3.4 Results of SE

SE is a key indicator to verify if grouts is adaptive to flowing water grouting. A widely used calculation method was used in this work to evaluate the SE of nano-$CaCO_3$ composited grouts, as shown in Eq. (1) (Sui et al., 2015):

$$SE = (Q_0 - Q_g)/Q_0 \times 100\%, \tag{1}$$

where Q_0 is the initial water flow rate prior to grouting; Q_g is the water flow rate after grouting; SE is SE of grouts (Liu et al., 2020). The SE of grouts with various components were shown in Fig. 7.

The SE of ordinary grouts without nano-$CaCO_3$ was much smaller than that of nano-$CaCO_3$ based cement grouts. The SE of grouts (0.7 W/C) increased with nano-$CaCO_3$ when the nano-$CaCO_3$ was smaller than 1%. Then the SE decreased with an increase of nano-$CaCO_3$ content when the nano-$CaCO_3$ exceeded a certain value. The effect of nano-$CaCO_3$ on the SE was similar to that of MFP, suggesting that MFP had a good consistence with SE.

Flowing water grouting in rock fracture is a complex process. Grouts with too high viscosity will concentrate at grouting hole and can't diffuse across the whole fracture, causing the failure of grouting. On the other hand, the low viscosity and low early strength of grouts will result in the low flowing water resistance of grouts. Then grouts will be scatter and washed out with little retention in fracture (Yang et al., 2018). Therefore, the viscosity and early strength of grouts have a combined effect on the SE of

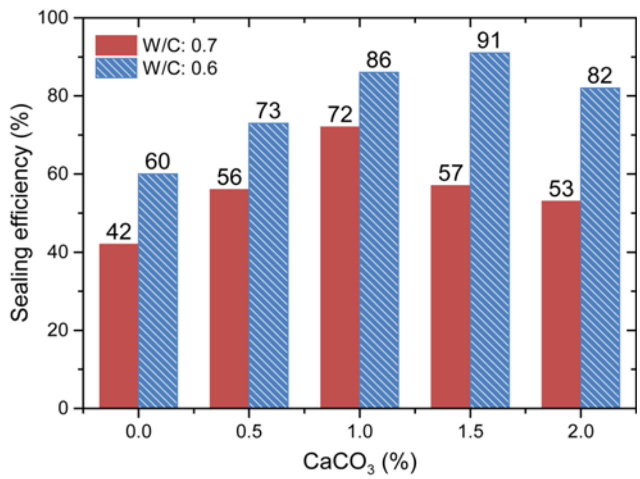


Fig. 7. SE of Grouts in Flowing Water Grouting Simulation Test

grouting. When the content of nano-$CaCO_3$ was less than 1%, early strength and flowing water resistance of grouts with 0.7 W/C increased rapidly and the viscosity had little variation with an increase of nano-$CaCO_3$, resulting in the increase of SE. When nano-$CaCO_3$ exceeded 1%, viscosity of grouts increased obviously (Fig. 3) and the negative influence of viscosity was greater than the positive influence of the increase of early strength, resulting in the decrease of SE.

Additionally, the SE of grouts with 0.6 W/C and 40% fly ash was much greater than SE of grouts with same nano-$CaCO_3$ content and 0.7 W/C. The decrease of W/C can increase the strength and fly ash can enhance the flowability of grouts. Therefore, reducing W/C and mixing with fly ash was an effective method to increase the SE of grouts. It's worth noting that nano-$CaCO_3$ can only improve the early strength of grouts and had little effect on the strength of cement in late stage (Meng et al., 2017). However, Meng et al. (2017) proved that fly ash can improve the strength of cement with nano-$CaCO_3$ in late stage due to the pozzolanic reaction of fly ash. Although the influence of strength of grouts in late stage was smaller than that of early strength, greater strength of grouts in late stage was helpful to the stability of rock fracture sealing. When the grouts composed of 1.5% nano-$CaCO_3$, 40% fly ash and 0.6 W/C, the SE was highest which reached 91%.

3.5 Discussion on the Application in Practical Engineering

The research results provide a new feasible way to improve effectiveness of flowing water grouting by mixing cement based grouts with nano-$CaCO_3$ and fly ash. Engineers can adjust the grouting parameters, e.g., nano-$CaCO_3$ content, W/C, and fly ash content according to results of simulation tests and natural condition of practical engineering. For example, nano-$CaCO_3$ can be increased to enlarge the viscosity and MFP when the flowing water is great or the fracture is inclined. Then the adverse effect caused by great flowing water or inclination can be eliminated and the SE will be improved.

Compared with other nanomaterials, nano-$CaCO_3$ is more applicable to practical engineering due to its low cost. The combined application of nano-$CaCO_3$ and fly ash can decrease the cost of grouting in further, which is crucial to flowing water grouting in practical engineering. This advantage enlarges when the grouting amount is great. The optional composition of grouts acquired in this work provides a reference for the selection of grouting parameters in practical engineering, overcoming the disadvantage of selecting grouting parameters by experience.

4. Conclusions

A new set-up was created to simulate grouting in flowing water condition in rough fracture. Effects of nano-$CaCO_3$ on the viscosity, microstructure, fluid pressure and SE of grouts were studied in this work. Increasing nano-$CaCO_3$ resulted in a decrease of grouts viscosity due to an increase of free water and the disturbance on additive. Compared with grouts without nano-$CaCO_3$, the MFP and SE of grouts with nano-$CaCO_3$ increased obviously. The MFP and SE increased in the initial stage with an increase of nano-$CaCO_3$, because nano-$CaCO_3$ can improve the early strength and microstructure of grouts. When the nano-$CaCO_3$ content exceeds a certain value, the MFP and SE decreased with an increase of nano-$CaCO_3$ due to the decrease of viscosity. SEM tests suggest that nano-$CaCO_3$ can enhance the flowing water resistance of grouts through forming fibrous hydrates, and fly ash can enhance the fluidity of grouts through ball effect. The SE of grouts with 0.6 W/C and 40% fly ash was greater than that of grouts with 0.7 W/C, indicating that reducing W/C and incorporating fly ash was an effective method to improve grouting effectiveness. The composition of 0.6 W/C, 40% fly ash and 1.5% nano-$CaCO_3$ was optimal composition of grouts with highest SE (91%) in this work, providing significant references to the practical engineering.

Acknowledgments

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