International Journal of Civil Engineering

Geotechnique

Technical Note

Pore structures and mechanical properties of microbe-inspired cementing sand columns

L. Li[1,]*, Q. Chun-xiang[2], Z. Yong-hao[3]

Received: December 2012, Revised: November 2013, Accepted: January 2014

Abstract

It introduced an innovative bioengineering method of consolidating incompact sand by urea-hydrolysis producing calcite cementation under the inducement of urease producing microbe. In the paper it discussed the effects of cementation methods and time on porosity and mechanical properties of microbe-inspired cementing sand columns. Method A adopted reaction fluid gravitational permeating and external pressing and method B adopted reaction fluid gravitational permeating and outlet intermittent plugging method. 28-day sand columns prepared by method A exhibited stronger mechanical properties than those prepared by method B, considering of the compressive strengths and three-point flexural strength as well. Pore volume fractions of sand columns prepared by method A reduced with an increase in cementation time which represented the bulk densities of sand columns were improved positively with time. The compressive strengths and the flexural strengths of sand columns prepared by method A increased with time. All these improved mechanical properties were attributed to the fact that the increasing amount of microbe inspired calcite precipitation with time consolidated sand columns by filling or bridging in sand gaps.

Keywords: *Microbe-inspired cementation, Sand column, Mechanical property, Porosity, Permeability.*

1. Introduction

Microbes, through their own living activities, take an important part in the environment with their enzyme. With a necessary reactive time, microbes help to consolidate the incompact clastic sand and stones into large-size hard rocks. Those rocks are mostly made of calcite-like materials. This kind of microbes is called urease-producing bacteria, or carbonate-mineralizing bacteria. Urease-producing microbe works as a catalyst in urea hydrolysis and calcite precipitation [1]-[3]. This particular carbonate precipitation process is called Microbial Inspired Calcite Precipitation (MCP, as abbreviation later), as function (1) and (2) [4]-[7].

$$CO(NH_2)_2 + 2H_2O \rightarrow CO_3^{2-} + 2NH_4^{+} \quad (1)$$
$$Ca^{2+} + CO_3^{2-} \rightarrow CaCO_3\downarrow \quad (2)$$

In the paper it applied two cementation methods both based on MCP mechanism to cement incompact sand into sand columns and investigated the relationships of porosity, compressive strength and three-point flexural strength with cementation methods and time.

2. Experimental Procedure

2.1. Microbe inspired sand cementation methods

Sand of 15 g in 200 μm diameter was added into a plastic injection tube of 22 mm in diameter and 95 mm in length with an appropriate vibration. Those tubes filled with sand were prepared for microbe inspired calcite cementation.

Two completely different methods were invented to execute on sand cementation. Method A adopted reaction fluid gravitational permeating and external pressing while method B adopted reaction fluid gravitational permeating and outlet intermittent plugging. They were specifically described as following. Sand columns were divided into 6 groups, in Table 1.

** Corresponding author: lili_smse@njust.edu.cn*

1 Postdoctor, Nano-structural Materials Center, School of Materials Science and Engineering, Nanjing University of Science and Technology, Street of Xiao Ling Wei 200, Nanjing 210009, Jiang Su Province, China

2 Professor, East of Ji Yin Da Dao, Jiangning, School of Materials Science and Engineering, Southeast Unvi., Nanjing 211189, Jiang Su Province, China

3 Professor, Executive Director, Nano-structural Materials Center, School of Materials Science and Engineering, Nanjing University of Science and Technology, Street of Xiao Ling Wei 200, Nanjing 210009, Jiang Su Province, China

Table 1 Experiment design of MCP cementation

Type	Method		Fluid components	Time /d
	Pressing	Plugging		
A1～A6	Y	N	Bacteria、$CaCl_2$、urea	5
A7～A12	Y	N	Bacteria、$CaCl_2$、urea	10
A13～A18	Y	N	Bacteria、$CaCl_2$、urea	15
A19～A24	Y	N	Bacteria、$CaCl_2$、urea	20
A25～A30	Y	N	Bacteria、$CaCl_2$、urea	28
B1～B6	N	Y	Bacteria、$CaCl_2$、urea	28
C1	Y	N	Water	-
C2	N	N	Water	-

2.1.1. Method A

It was a method of reaction fluid gravitational permeating and external pressing. The characteristic of method A was to make the sand compact as much as possible under 0.1 MPa axial pressures during the whole cementation. An iron frame was designed and made to allow tubes located in it tightly and easily, Fig.1 and Fig.2.

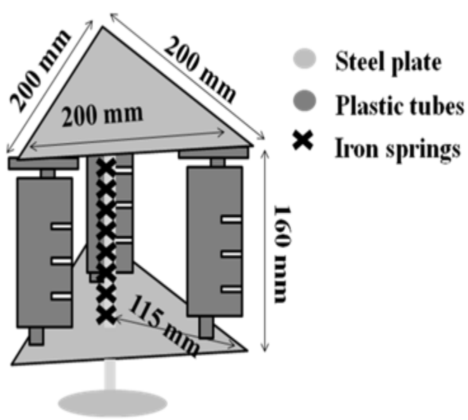
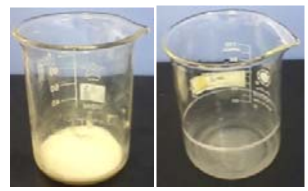


Fig. 1 Steel set-up schematic diagram of Method A

Fig. 2 Photos of MCP cementation of Method A

2.1.2. Method B

It was a method of reaction fluid gravitational permeating and outlet intermittent plugging. Compared with method A, method B differed in no pressure on sand but outlet intermittent plugging. The characteristic of method B was to make the calcite stay in sand as much as possible at each outlet plugging time, which was proved by the amount of calcite precipitated on the beakers' bottom to collect the out-flowing fluid from outlet, Fig.3.



(a) (b)

Fig. 3 Calcite precipitation at beaker's bottom (a) method A (b) method

2.2. Mercury intrusion porosimetry method

Porosimetry is an analytic technique used to determine various quantifiable aspects of a material's porous nature, such as pore diameter, total pore volume, superficial area and pore throat ratio. The MIP technique involves the intrusion of a non-wetting liquid (often mercury) at high pressure into a material through the use of porosimeter. The pore size can be determined based on the external pressure needed to force the liquid into a pore against the opposing force of the liquid's superficial tension.

The sizes and size distributions of pores and throats and their arrangement relative to each other are determined from mercury intrusion-extrusion capillary pressure curves. And the pore-throat aspect ratio is the ratio of pore radius to the linked throat radius, which indicates their dimensional arrangement in material, Fig.4.

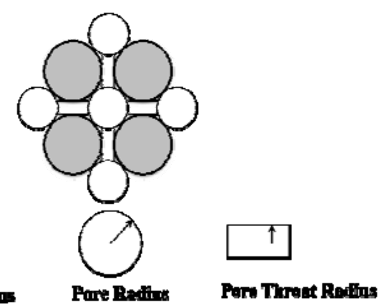


Fig. 4 2-D sand, pore and pore throat radius

2.3. Compression test

Compressive strength worked out from compression test results is the capacity of a material to withstand axially directed pushing forces. When the limit of compressive strength is reached, materials are crushed. When a material gets a compression, it gets shorter which seemly tends to amplify small sideways deflections into buckling. A material such as soft sandstone may have a compressive strength as low as 1 MPa or 5 MPa. Here is a basic definition of the uniaxial stress given by equation (3).

$$\delta_c = F / A \quad (3)$$

Where, δ_c is the uniaxial stress, MPa; F is the load applied, N; A is the area the load applied on, mm^2.

2.4. Three-point flexural test

The objective of Three points flexural test is to provide an efficient and consistent methodology to locate and characterize fracture origins in advanced ceramics. It is applicable to brittle materials like ceramics; that is, the material adheres to Hooke's Law up to fracture. In such materials, fracture commences from a single location which is termed the fracture origin. The fracture origin in brittle ceramics normally consists of some irregularity or singularity in the material which acts as a stress concentrator. In the parlance of the engineer or scientist, these irregularities are termed "flaws" or "defects." Equation (4) is for a calculation of the flexural stress for a circular cross section, based on ASTM C1322-96a, Standard practice for fractography and characterization of fracture origins in advanced ceramics.

$$\delta_f = PL/(\pi R^3) \quad (4)$$

Where, δ_f is the stress at midpoint, MPa; P is the load at a given point on the load deflection curve, N; R is the cross section diameter of the specimen, mm; L is the span length of the specimen.

3. Results and Discussion

3.1. Porosity and Pore-throat aspect ratio

It was shown in Fig.5 where MIP samples were taken.

A lot of factors' data were obtained from the porosity standard report of MIP, such as permeable area, total porosity volume, total porosity volume fraction, specific superficial area and pore-throat aspect ratio, which were listed in Table 2. The relationship between total porosity volume and cementation methods and time and the relationships between pore-throat aspect ratio and cementation methods and time were shown in Fig 6.

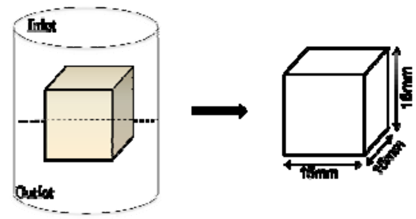


Fig. 5 MIP samples taken from column

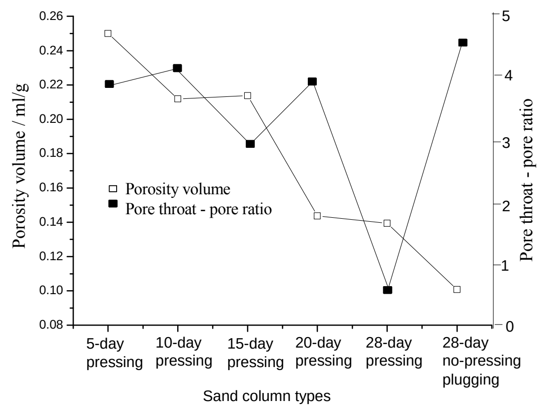


Fig. 6 Porosity volume and pore-throat aspect ratio of sand columns of different cementing methods at ages

Referring to the permeable area, the rock permeability grades were shown in Table 3. Compared with the permeable area of grades in Table 3 [8], all the sand columns prepared by method A fell into the super low grade.

Table 2 Calculated porosity structure factors of MIP data report of sand columns

No.	Days /d	Grades	Permeable area /$10^{-3}\mu m^2$	Total porosity/%	Pore volume /ml/g	Specific area /m^2/m	Pore throat/pore
A1	5	Super low	0.08356	40.466	0.2500	0.0166	3.8286
A9	10	Super low	0.11084	35.724	0.2119	0.0169	4.1489
A13	15	Super low	0.03961	35.538	0.2137	0.0186	2.8909
A20	20	Super low	0.01479	27.855	0.1437	0.0236	3.8554
A27	28	Super low	0.00044	27.305	0.1394	0.1170	0.4865
B1	28	Super low	0.01210	18.183	0.1008	0.0079	4.6848

Table 3 Permeability grades of rocks

Grades	1	2	3	4	5	6	7
Permeability	Super high	Very high	High	Medium	Low	Very low	Super Low
Permeable area /$10^{-3}\mu m^2$	>1000	1000～500	500～100	100～50	50～10	10～1	<1

The total porosity fraction of sand columns of cementation time of 120 hours, 240 hours, 360 hours, 480 hours and 672 hours were 40.5%, 35.7%, 35.5%, 27.9% and 27.3%. The rule here were obvious that total porosity fraction reduced with an increase in cementation time which represented the bulk densities of sand columns were improved positively with increasing cementation time.

Although there was no exactly linear relativity between pore-throat aspect ratios and cementation time, pore-throat aspect ratio of sand columns at 672 hours sharply decreased to 0.49 from 3.83 at 120 hours. Based on the discussion earlier the pore-throat aspect ratio indicated dimensional arrangement of pores and throats, it explained to the MIP results that pore radius decreased when more and more calcite precipitation formed and filled into the pores with increasing cementation time.

3.2. Compressive strength

SANS CMT 8502 Computer Assisted Material Test Machine was applied to execute the compression test on sand columns. The load range of the machine was from 20 N to 5 KN and the head moved to the sand column at a speed of 0.5 mm/s. There was a revising coefficient of compressive strength related directly to the length-diameter ratio of sand columns which in this investigation the coefficient was 0.93 [9]. In order to clarify the relationships between compressive strength and cementation methods or time, data were plotted in Fig.7 and Fig.8.

The compressive strengths of cementation time of 240 hours, 360 hours, 480 hours and 672 hours after timing with 0.93 the revising coefficient were 0.56 MPa, 0.68 Pa, 1.20 MPa and 1.40 MPa, respectively. There was a clear relationship between the compressive strengths and the cementation time, which was compressive strengths increased with time since the increasing amount of calcite with time strengthened the sand columns obviously. The reason for the missing data of 120 hours of compression and flexural was all the sand columns at this age were unexpectedly broken up while demoulding.

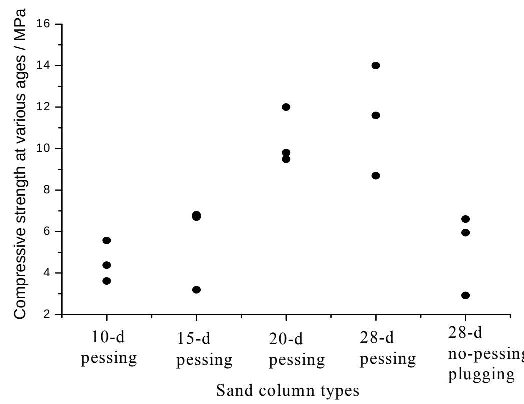


Fig. 7 Compressive strength of sand columns at various ages

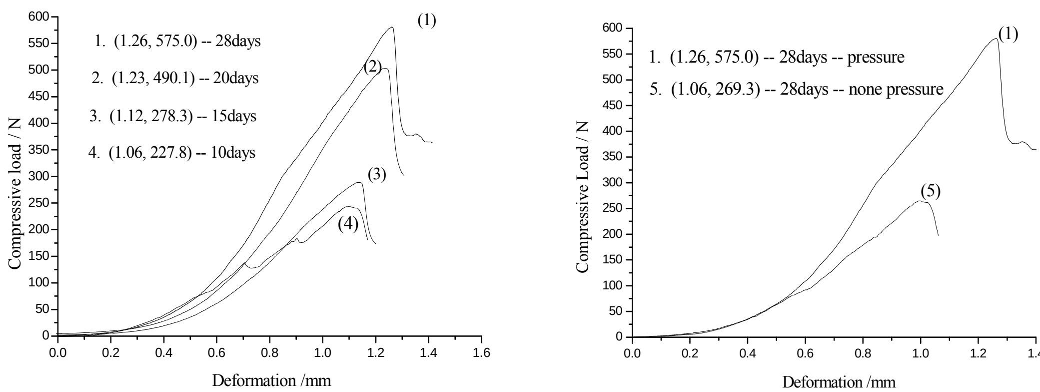


Fig. 8 Ages and cementing methods effect on compressive load — deformation curves ages (b) cementing methods

3.3. Three-point flexural strength

SANS CMT 8502 Computer Assisted Material Test Machine was applied to execute the three-point flexural test on sand columns. The load range of the machine was from 20 N to 5 KN and the head moved to the sand column at a speed of 0.5 mm/s. Six different testing modes were ready for options. The one adopted was the mode for engineering ceramic pipes.

The data of loads and deformations during flexural were automatically plotted by the computer. All the graphs were organized up into two sets. One differed in cementation time and the other did in cementation methods, shown in Fig.9 and Fig.10 respectively.

The flexural strengths of cementation time of 240 hours, 360 hours, 480 hours and 672 hours were 0.08 MPa, 0.13 Pa, 0.84 MPa and 0.96 MPa, respectively. The strengths increased rapidly with time. To be more observing, the bouncing on the up part of the load-deformation curves shrank, which theoretically indicated the defects in sand column became less, and the up part of curves smoothed out with the cementation time increased [10]. These mechanical appearances originally were attributed to the fact that the increasing amount of calcite with time remedied the defects by filling or bridging in sand gaps.

The flexural strengths of cementation method A and B were 0.96 and 0.41 MPa. The deformations at the peak loads of them were 1.29 mm and 0.80 mm. And the bouncing of method A columns were a lot more than the ones of method B. Conclusively the sand columns prepared by method A mechanically performed much better than those by method B because external pressure in method A cooperated with the inner strength development under MCP cementation.

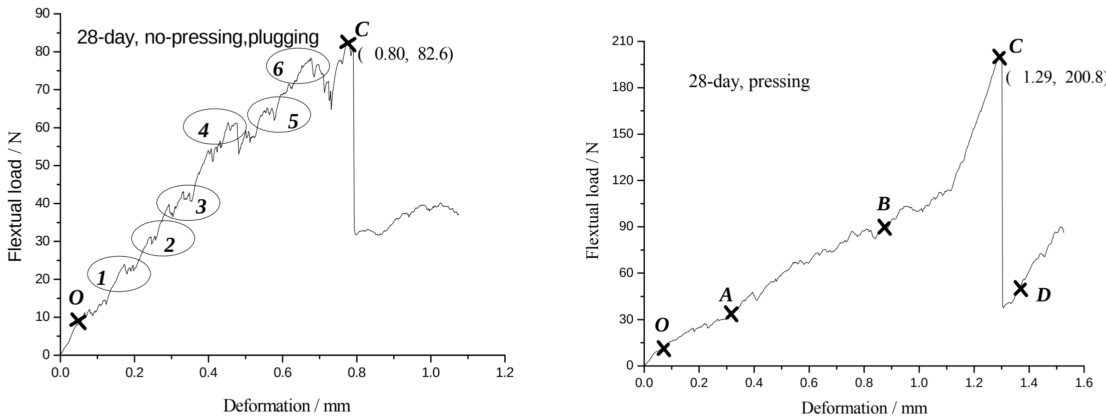


Fig. 9 Load-deformation curves of sand columns with different cementing methods pressing (b) no-pressing

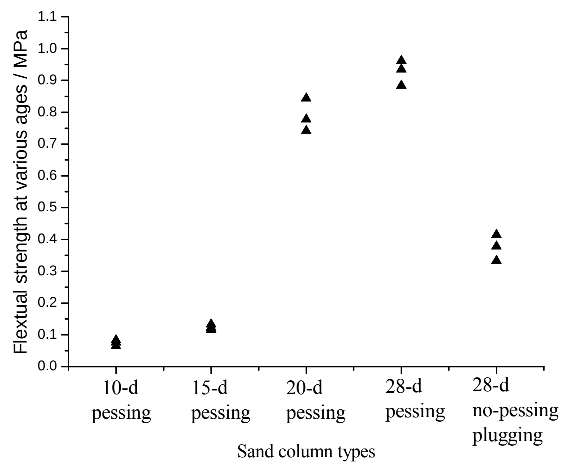


Fig. 10 Three-point flexural strength of sand columns at various ages

4. Conclusions

What the main purpose of the investigation was to discover the effects of cementation methods and time on mechanical performance and porosity of sand columns cemented by MCP technique. Here were the discoveries.

28-day sand columns prepared by method A exhibited stronger mechanical properties than those prepared by Method B, considering of the compressive strengths and three-point flexural strength. It could be exactly explained by porosity examination results that the permeable area of the 28-day columns by method A were extremely lower than those of the ones by Method B, which indicated that 28-day columns by method A were more solid and compact than those of the ones by method B.

In consideration of sand columns prepared by method A, pore volume fractions of them reduced with an increase in cementation time which represented the bulk densities of sand columns were improved positively with time. And there was a clear relationship between the compressive strengths and the cementation time, which was compressive strengths increased with time since the increasing amount of calcite with time strengthened the sand columns obviously. Also the flexural strengths increased rapidly with time. Both mechanical performances should attribute to the fact that the increasing amount of calcite with time remedied the defects by filling or bridging in sand gaps.

Acknowledgement: Authors are thankful to Jiangsu Planned Projects for Postdoctoral Research Funds.

References

[1] Reid R P, Visscher P T, Decho A W. The role of microbes in accretion, lamination and early lithification of modern marine stromatolites, Nature, 2000, No. 3, Vol. 406, pp. 989–992.

[2] Whiffin V, Harkes M, Paassen L. Microbial Carbonate Precipitation as a soil improvement technique, Geomicrobiology Journal, 2007, Vol. 14, pp. 417-423.

[3] Dick J, Windt W, Graef B. Bio-deposition of a calcium carbonate layer on degraded limestone by Bacillus species, Biodegraded Radiation, 2006, Vol. 17, pp. 357-367.

[4] Wang R.X, Qian C.X, Wang J.Y. A pilot study of microbial inducing calcium carbonate precipitation, Journal of Southeast University (nature science version), 2005, No. 4, Vol. 7, pp. 192-195.

[5] Dejong J. Microbially induced calcite cementation to improve soil behavior, Sedimentary Geology, 2007, No. 4, Vol. 197, pp. 769-773.

[6] Shu M, Morou-bermude E, Suarez-Perez E. The relationshiip between urease carriers and urease activity, Microbiology, 2007, No. 1, Vol. 22, pp. 61-66.

[7] Geotech J, Geoenvir E. Urease effects on urea hydrolysis, Biological Considerations in Geotechnical Engineering, 2005, No. 10, Vol. 131, pp. 1222-1233.

[8] Cooke A J, Rowe R K, Rittmann B E. Modelling species fate and porous media effects for landfill leachate flow, Canadian Geotechnical Journal, 2002, No. 1, Vol. 42(1), pp. 1116-1132.

[9] Pana P.Z, Fenga X.T, Hudson J.A. Study of failure and scale effects in rocks under uniaxial compression using 3D cellular automata, International Journal of Rock Mechanics and Mining Sciences, 2009, No. 4, Vol. 46, pp. 674-685.

[10] Pancheri P, Bosetti P, Maschio R D. Production of sharp cracks in ceramic materials by three-point bending of sandwiched specimens, Engineering Fracture Mechanics, 2008, No. 4, Vol. 59, pp. 447-456.