Experimental Assessment of Mechanical and Hydraulic Performance of Cement Sealing in Rock Salt

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Abstract

Frictional shear strengths of cement sealing in rock salt have been experimentally determined by series of borehole push-out testing and direct shear testing. The results are used to assist in design of the cement seals in the rock salt to minimize brine circulation and potential leakage along a main access of salt mine. The salt specimens are prepared from 100 mm diameter cores drilled from Middle member of the MahaSarakham formation. The cement seal is prepared from commercial grade Portland-pozzolan cement, saturated brine, anti-form agent and liquid additive. The cement slurry is casted in the 25 mm diameter borehole with a length of 30 mm for the push-out testing and on the 100 mm diameter fracture saw cut surface for the direct shear testing. For all tests the cement is cured for 7 days prior to testing. The results indicate that dynamic viscosity of grout slurry is 4.53 Pa.s. When the curing time increases the intrinsic permeability of cement grout decreases. The uniaxial compressive and Brazilian tensile strengths after 28 day curing times are 20.06±3.82 MPa and 2.89±0.19 MPa, respectively. The direct shear tests results indicate the frictional resistance at cement-salt interface with a friction angle of 44 degrees and a cohesion of 2.12 MPa. The normal stiffness is 7.67 GPa/m. The shear stiffness is 6.60 GPa/m. The push-out test results show significantly the higher frictional resistance at the interface than does the direct shear testing. The axial shear strength of the borehole cement seal is 5.05 MPa. The findings are useful for determination of initial installation parameters of the cement seals in salt mine openings.

Keywords: Rock salt,Sealing, Push-out test, Direct shear test, Permeability

1. Introduction

Design of seals may also be required in sealing mine openings in order to control mine effluents. Generally, cementing materials are used to seal fractures and dissolved channels to prevent brine leakage along the openings. The primary function of borehole and shaft seals is to reduce hydraulic conductivity of the openings to an acceptable level. Axial load on plugs or seals in an underground salt mine leads to shear stresses along a contact between the plug and a host rock. These shear stresses may cause cracking and increased permeability along the cement plug and rock interfaces. Under extreme condition they can cause slipping of the cement plugs. Therefore, a bond between plug and rock is a critical element of the design and performance of plug in the excavated opennings (Fuenkajorn and Daemen, 1996; Akgun and Daemen, 1997; Tepnarong, 2012).

The objectives of this study are to determine the bond strength of cement grout borehole plugs cast in rock salt cylinders through the push-out testing and to determine the shear strength between cement-salt interfaces by the direct shear testing as a function of cement curing period. The results are used to assist in along-term design of the cement seals in fracture and dissolved channels to minimize a brine circulation and potential leakage along a main access of a salt mine in the northeast area of Thailand.

2. Rock salt specimens

100 mm diameter core samples of rock salt are donated by Asian Potash Mining Company. They are collected from a borehole located in Bumnetnarong district, Chaiyaphum province. All sample are obtained from the Middle Salt member at depths between 70 m and 130 m of theKhorat basin. The petrographic properties of the tested cores are as follows. The salt crystals are tightly interlocked. The white and clear halite change to pale, medium and dark honey when the depths increase. The diameters of milky white halite range from 0.1 to 0.3 cm, showing flow texture. Smoky dark halite is associated with anhydrite. The large recrystallized glassy halite is found interlayered with the honey

halite. The associated minerals include local occurrence of sylvite and carnallite. Some large crystals ranging from 2.0 to 5.0 cm are locally occurred. (Tepnarong, 2012).

Sample preparation are followed the ASTM D4543 standard practice, as much as practical. Specimens are prepared for the direct shear strength tests and the push-out tests with length of 100 mm. The cylindrical push-out specimens are drilled with 25 mm diameter holes perpendicular to the bottom sample surfaces (Fig.1). The saw cut surfacesare prepared for the direct shear testing. After preparation, the specimens are labeled and wrapped with a plastic film. The specimen designation is identified.

Fig. 1.Examplesof salt specimensprepared for push-out test.

3. Cement grout preparation

The cement mixing for the borehole plugs and the direct shear testing specimens is performed according to the API No. 10 (American Petroleum Institute, 1986; AkgunandDaemen, 1997) by mixing cement with a salt (NaCl) saturated brine. The components of cement slurry are commercial grade Portland cement mixed with chloride resistant agent, NaCl saturated brine, anti-form agent and a liquid additive including expansion. The brine is prepared by dissolving clean rock salt in distilled water. Portland-pozzolan cement is chosen due to its low brine demand, sulfate resistance and widely used in construction industry. A liquid additive contributes to expansiveness of mixture. An anti-forming agent is used to decrease the air content of the cement slurry and to ease a control of cement slurry weight and volume. Table 1 gives the weight compositions of the mixture. The saturated brine is poured into a mixing container at low speed, and all components are added to the brine within 15 seconds. After all the cement is added, the slurry is mixed at high speed for additional 35 seconds. The cement grout preparation is cured under atmospheric pressure, at room temperature (28 to 30°C).

Table 1 Weight composition of grouting cement slurry.

Material	Weight (g)
Portland-pozzolan cement, type IP	1000
NaCl saturated brine	670
Liquid additive	10
Anti-form agent	10

4. Mechanical characterization of cement grout

Characterization testing provides the uniaxial compressive strength (σ_c), Young's modulus (E), Brazilian tensile strength (σ_B) and viscosity and slurry density of cement grout. Specimens of 54 mm diameter cylindrical cement specimens with length to diameter ratios between 2.5 and 3.0 are prepared by curing cement pastes in PVC molds for 1, 3, 7, 14, 21 and 28 days. They are cured at room temperature (ASTM C192). Each mold is puddled with the puddling rod to eliminate cement segregation. The remaining portion of the molds is filled with brine. All cement cylinders are taken out of their molds after each curing period. The ends of specimen are cut and paralleled.

The uniaxial compressive strength test procedure is followed, as much as practical, the ASTM standards (D7012 and C39). The cement grouts are loaded at the constant rate of 0.1-0.5 MPa/s until failure. The axial displacements are monitored by displacement dial gauges. Table 2 shows the average compressive strength and the elastic modulus as function of the curing time.

The Brazilian tensile strength tests are performed in accordance with ASTM standard (D3967). The load is applied along the diameter of the specimen until failure occurred. The average and standard deviation of characterization tests are shown in Table 2.

Viscosity measurement follows, as much as practical, ASTM D2196. The viscosity is measured with Brookfield® viscometer. The average dynamics viscosity of cement slurry is 4.53 Pa·s. The average kinematic viscosity of cement slurry is 2.59×10^{-3} m^2/s. The average cement slurry density is determined as 1.75 g/cc.

Table 2 Uniaxial compressive strength, elastic modulus and Brazilian tensile strength of the cement plugs.

Specimen Age (day)	Average Density (g/cm^3)	σ_c (MPa)	E (GPa)	σ_B (MPa)
1	1.73±0.01	3.32±0.04	0.92±0.10	0.44±0.01
3	1.72±0.01	9.45±1.25	1.32±0.19	1.54±0.06
7	1.73±0.01	14.24±2.16	1.25±0.12	1.59±0.08
14	1.72±0.01	18.42±2.53	1.64±0.51	1.88±0.13
21	1.73±0.02	18.64±1.23	1.29±0.01	2.36±0.28
28	1.74±0.01	20.06±3.82	1.79±0.58	2.89±0.19

5. Push-out test

The objective of this test is to determine the axial mechanical strength and long-term deformation of borehole plugs in rock salt core through push-out tests. The curing period for push-out tests is 7 days. Fig.2 shows the schematic drawing of the push-out test setup. A cylindrical steel rod applies an axial load to a cement plug. The top and bottom displacement of the borehole plug are measured by dial gages. The axial load is measured by a load gage of hydraulic pump. The displacement is measured manually by dial gages with a resolution of 0.025 mm. A loading frame with a hydraulic cylinder applies the load. The machine has a capacity of 50 kN with a resolution of 0.5 kN.

Fig.3 shows the push-out test setup. A salt cylinder in PVC molds with cement plug is centered on the square platen. A steel cylinder of slightly smaller diameter than the plug is centered and transmits the load on top of the plug. The specimen is loaded under constant stress rate of 0.1 MPa/second. The load and top and bottom plug displacements are recorded manually at 10 seconds intervals until sliding occurs.

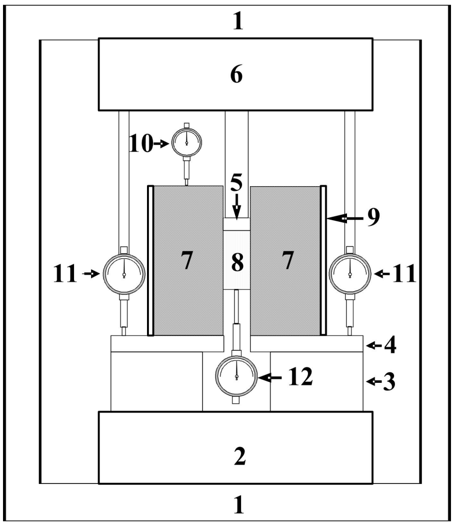

Fig. 2.Schematic drawing of the push-out test setup 1.Loading frame; 2.Hydraulic cylinder; 3.Steel plate with a slit; 4.Square steel plate; 5.Axial bar and steel cylinder; 6.Square steel plate frame; 7.Rock salt sample; 8. Cement grout plug; 9. PVC mold; 10., 11. and 12. Dial gages.

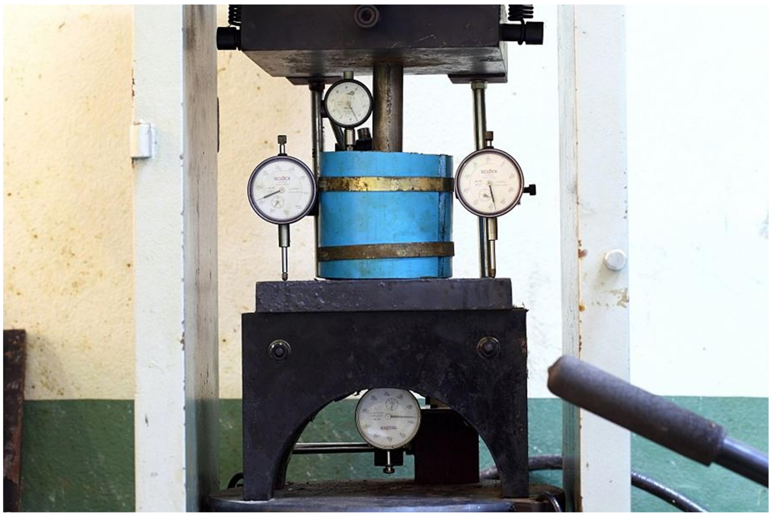

Fig. 3.The push-out test setup.

The bond strength or the average shear stress (τ_{av}) distribution induced by push-out test loading along the rock salt/cement plug interface can be calculated by the following equation (Stormont andDaeman, 1983):

$$\tau_{av} = F / \pi D_i L_c \tag{1}$$

where$F$ is the failure load, D_i is the plug diameter andL_c is the plug length.

Fig.4 plots the applied axial stress as a function of the top and bottom plug displacements. The bottom plug displacements are small compared to the top axial displacements prior to bond failure. Upon a plug slip, the difference between the top and the bottom plug displacements decrease most probably due to stress relief caused by slip along the interface. Rock bridges fail at the applied axial stresses at 22.48 MPa. The bond strength is 5.05 MPa.

The push out tests are performed on cement plugs with a series of relatively long curing time with a constant shear stress. Fig. 5plots the top plug displacements as a function of time with a various constant shear stress. The results show an instantaneous elastic strain. Fig.6 shows sample no.SBIIH-04-07-PO-01 which is cut along the axis after failure. The thick cement residue on the borehole walls above the (slipped) cement plug and absence of dissolutioning indicate gooda bonding.

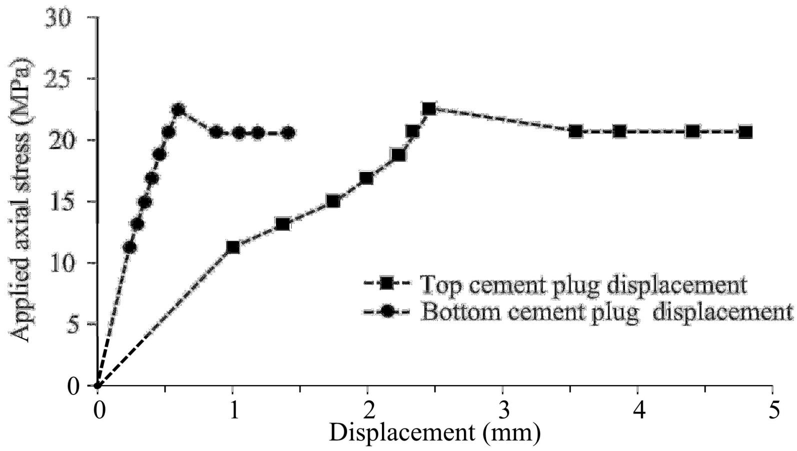

Fig. 4.The applied axial stress as a function of the top and the bottom axial cement plug displacements for the push-out test (specimen no.SBIIH-04-07-PO-01).

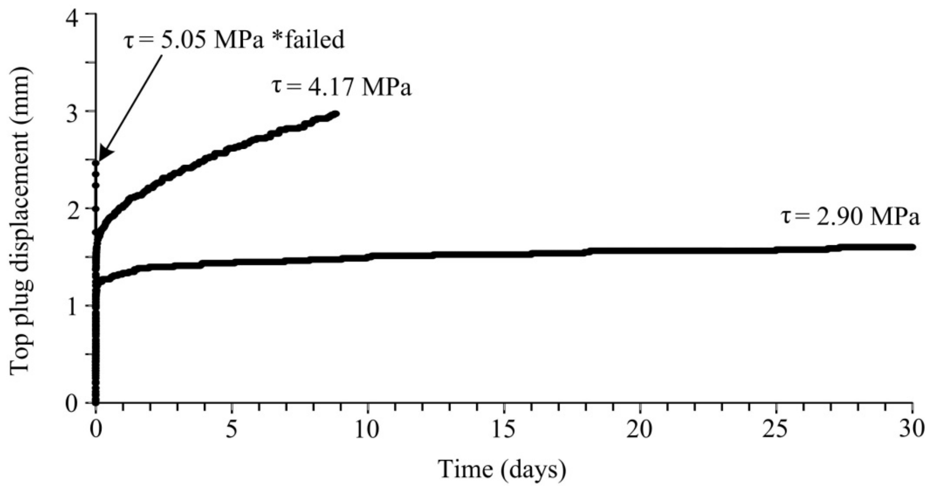

Fig. 5.The top plug displacement as a function of time for the push-out tests.

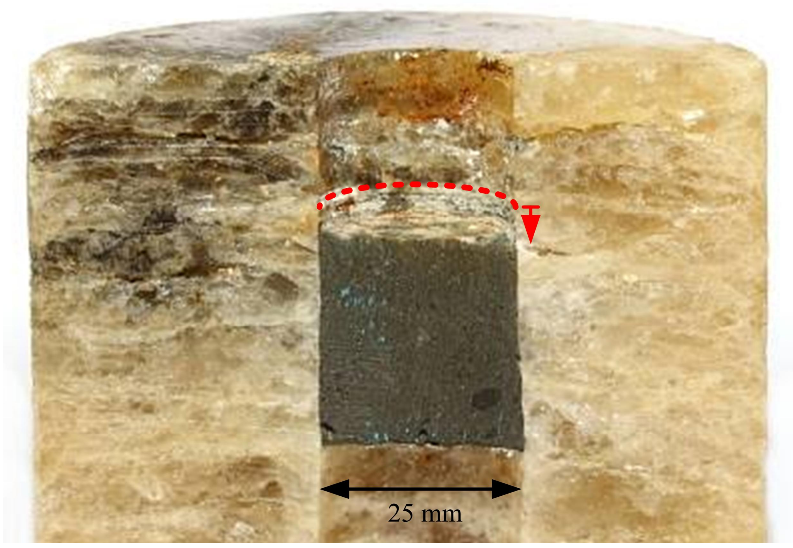

Fig. 6.A cut section of specimen no.SBIIH-04-07-PO-01 after failure in the push-out test.

6. Direct shear test

The shearing resistance between cement grout and rock salt fracture isdetermine by the direct shear testing.The test procedure is similar to the ASTM D5607 standard practice.The cement slurry is casted on 100 mm diameter saw cut fracture surfaces. The cement is cured for 7 days prior to testing for all tests. The direct shear machine model EL-77-1030 with both of the maximum shear load and normal load of 50 kNis used. Laboratory arrangement for the direct shear test equipment is shown in Fig.7. Pre-defined normal loads are maintained constant by during test. The constant normal stressesare 0.62, 1.26, 1.89, 2.52 and 3.14 MPa. Shear force is continuously applied and monitored for every 0.2 mm of the shear displacement. The peak and the residual shear stress are calculated and plotted against the corresponding normal stress. Linear relationship between shear and normal stresses is obtained.

According to the Coulomb criterion the peak and the residual friction angles at the cement-rock salt interface are 44° and 42°, respectively. The cohesion of peak shear strength is 2.12 MPa. The normal stiffness and shear stiffness are 7.67 GPa/m and 6.60 GPa/m, respectively (Fig.8).

The long-term shear behavior of the cement-rock salt interface at a constant normal stress is shown in Fig.9.The creep displacement is a function of both the normal stress and the shear stress of the fracture. Under a constant normal stress, the creep deformation is expected to increase as the shear is increased.

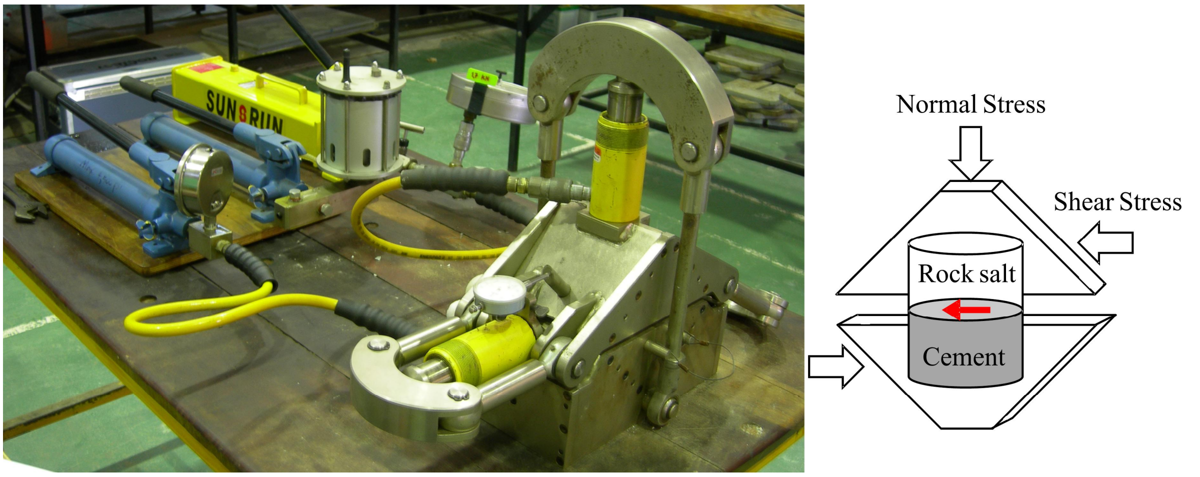

Fig.7. Laboratory equipment and instrumentation setup for the direct shear test.

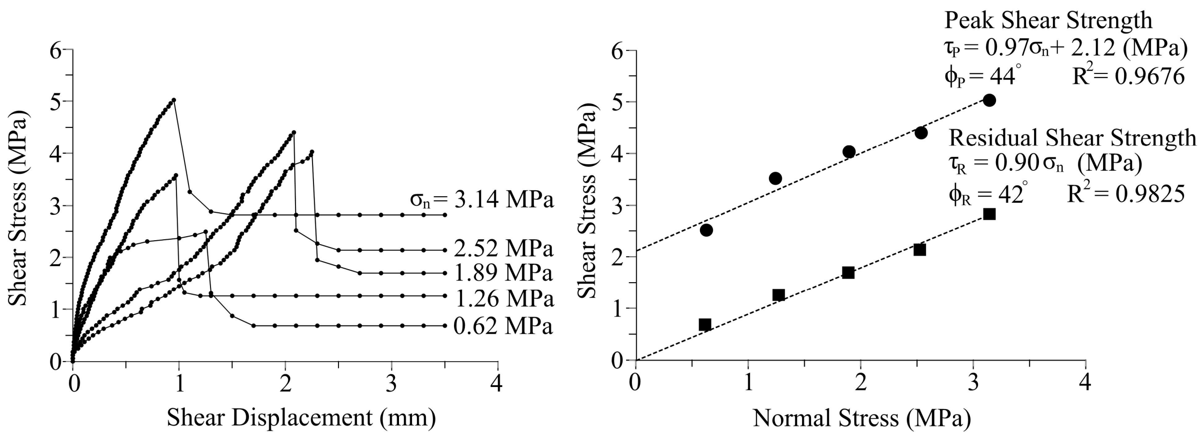

Fig. 8. Results of the direct shear testing.

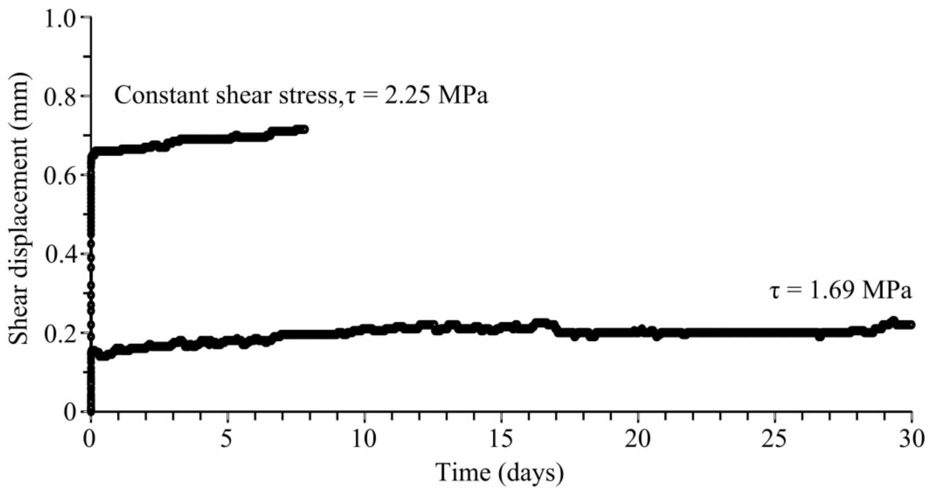

Fig. 9. Shear displacement as a function of time for the direct shear tests with a constant normal stress of 1.86 MPa.

7. Permeability of cement grout

The permeability of grouting materials is determined in term of the intrinsic permeability (k). The constant head flow test is conducted to measure the longitudinal permeability of the grout. Test pressure and specimen configuration are measured and used to calculate the coefficient of permeability. The permeability of a system considered herein is measured using a constant head apparatus as shown onFig.10. The flow in longitudinal direction of a tested system is described by Darcy's law.(Indraratna&Ranjith, 2001).The cylinder specimen is 100 mm in diameter and 100 mm long. After three days of curing, the specimen is carefully removed from the cast (PVC pipe), cleaned, and placed in a brine bath before installing in the permeability test apparatus. The permeability of the test system is measured and recorded at 1, 3, 7, 14, 21, 28, 35, 42 and 60 days of curing periods. The results indicate that when the curing time increases the intrinsic permeability (k) of cement grout decreases. The conductivity of permeability and intrinsic permeability of cement grouts as a function of curing time are shown in Fig.11.

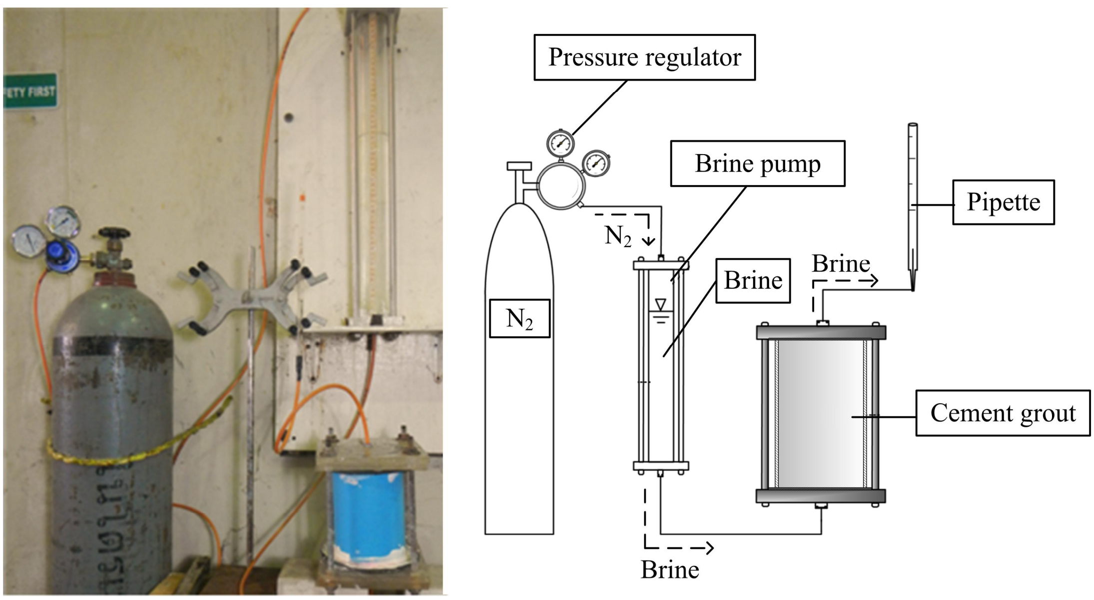

Fig. 10. Constant head flow test apparatus used for the permeability testing of a cement grout.

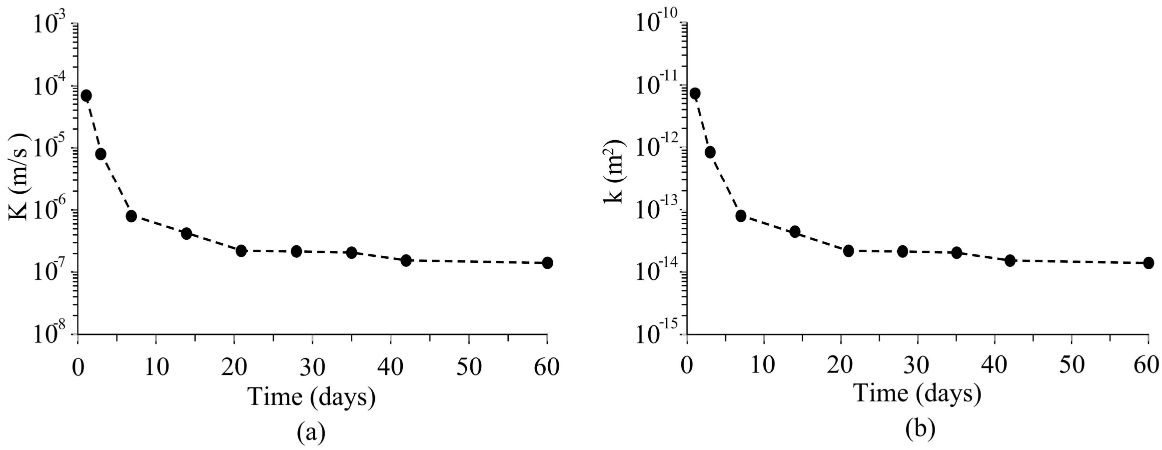

Fig. 11. (a) Conductivity of permeability (K) and (b) Intrinsic permeability (k) of the cement grouts.

8. Discussions and conclusions

Characterization test results indicate that when the curing time increases the uniaxial compressive strength, the elastic modulus, theBrazilian tensile strength of cement grout increases. The average dynamics and kinematic viscosity of cement slurry are 4.53 Pa·s and 2.59×10^{-3} m^2/s, respectively. The average cement slurry density is 1.75 g/cc.

The push-out tests and the direct shear tests are performed to determine the bond strength of cement plugs in the MahaSarakham rock salt. The cement slurry is casted in the 25 mm diameter borehole with a length of 30 mm for the push-out testing and on 100 mm diameter fracture saw cut surface for the direct shear testing. For all tests the cement is cured for 7 days prior to testing.

The direct shear tests results indicate the frictional resistance at cement-salt interface with the friction angle of 44 degrees and cohesion of 2.12 MPa. The normal stiffness is 7.67 GPa/m. The shear stiffness is 6.60 GPa/m.The push-out test results show significantly higher frictional resistance at the interface than does the direct shear testing. The axial shear strength of the borehole cement seal is 5.05 MPa. This is primarily due to the effect of the Poisson's ratio which increases the normal (radial) stress at the cement-salt interface while the axial load is applied. This implies that the direct shear test results may give an overly conservative estimate of the shearing resistance between the salt and cement seal.

The long-term permeability of the cement grouting materials measured from the longitudinal flow test with constant head decreases with curing time at 1, 3, 7, 14, 21, 28, 35, 42 and 60 days. The results indicate that when the curing time increases the intrinsic permeability (k) of the cement grout decreases.

Push out tests are performed on cement plugs with a series of relatively long curing time with the constant shear stress. The results plot the top plug displacements as a function of time with a various constant shear stress and show an instantaneous elastic strain. The long-term direct shear testing at a constant normal stress shows the creep displacement. Under a constant normal stress, the creep deformation is expected to increase as the shear is increased.

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References

Akgun, H. andDaemen, J.J.K., 1997, Analytical and experimental assessment of mechanical borehole sealing performance in rock, *Engineering Geology Journal*. 47: 233–241.

American Petroleum Institute, 1986, *Specifications for Materials and Testing for Well Cements. 3rd Edition*, American Petroleum Institute, Production Department, Dallas, TX.

ASTM Standard C192-07, 2007, Standard practice for making and curing concrete test specimens in the laboratory, *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, PA.

ASTM Standard C39-10, 2010, Standard test method for compressive strength of cylindrical concrete specimens, *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, PA.

ASTM Standard D2196-10, 2010, Standard test methods for rheological properties of non-newtonianmaterials by rotational (Brookfield type) viscometer, *Annual Book of ASTM Standard*, American Society for Testing and Materials, Philadelphia, PA.

ASTM Standard D3967-81, 1981, Standard test method for splitting tensile strength of intact rock core specimens, *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia. PA.

ASTM Standard D4543-85, 1985, Standard test method for preparing rock core specimens and determining dimensional and shape tolerances, *Annual Book of ASTM Standard*, American Society for Testing and Materials, Philadelphia, PA.

ASTM Standard D5607-08, 2008, Standard test method for performing laboratory direct shear strength tests of rock specimens under constant normal force, *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, PA.

ASTM Standard D7012-10, 2010, Standard test method for compressive strength and elastic moduli of intact rock core specimens under varying states of stress and temperatures, *Annual Book of ASTM Standards*, American Society for Testing and Materials, Philadelphia, PA.

Fuenkajorn, K. andDaemen, J.J.K. (Eds.), 1996*, Sealing of Boreholes and Underground Excavations in Rock,* Chapman and Hall, London.

Indraratna, B. and Ranjith, P., 2001, *Hydromechanical Aspects and Unsaturated Flow in Joints Rock*, Lisse: A. A. Balkema.

Stormont, J.C. andDaemen, J.J.K., 1983, Axial strength of cement borehole plugs in granite and basalt. *Tech. Rep. NUREG/CR-3594,* U.S. Nuclear Regulatory Commission, by theDepartment of Mining and Geological Engineering, University of Arizona, Tucson.

Tepnarong, P., 2012, Bond strength of cement sealing in MahaSarakhamsalt, *Proc. of 7th Asian Rock Mechanics Symposium*, Seoul, Korea, 15-19 October 2012, 584-593.