



EXPERIMENTAL ASSESSMENT ON BOREHOLE SEALING PERFORMANCE OF SLUDGE-MIXED  
CEMENT GROUT IN ROCK SALT

The Engineering Institute of Thailand under H.M. The King's Patronage

**EXPERIMENTAL ASSESSMENT ON BOREHOLE SEALING PERFORMANCE OF  
SLUDGE-MIXED CEMENT GROUT IN ROCK SALT**

Phadet Deethouw<sup>1</sup> and Prachya Tepnarong<sup>2</sup>

<sup>1</sup>Graduated Student, School of Geotechnology, Institute of Engineering, Suranaree University of Technology

<sup>2</sup>Lecturer, School of Geotechnology, Institute of Engineering, Suranaree University of Technology

**ABSTRACT**

*The objective of this study is to experimentally assess the performance of sludge-mixed cement grouts for sealing boreholes in rock salt. The cement grout is prepared from the commercial grade Portland cement mixed with Bang Khen water treatment sludge, brine and chloride resistant agent. The results are used in the design of borehole seal in rock salt to minimize the brine circulation and potential leakage for the industrial waste repository. The salt specimens are prepared from the 54 mm diameter cores drilled from the Middle member of the Maha Sarakham formation. The results indicate that the viscosity of grout slurry tends to increase as the sludge-mixed cement (S:C) ratio increases. The compressive strength after 28 day curing times is  $9.58 \pm 0.52$  MPa. The highest compressive strength is from S:C = 5:10. The average tensile strength is  $1.99 \pm 0.14$  MPa. The highest bond strength is 7.49 MPa. When the curing time increases, the intrinsic permeability of cement grouts decreases. Similarities and discrepancies of the grouting performance in terms of mechanical and hydraulic properties are compared.*

**KEYWORDS:** Sludge-mixed cement grout, Rock salt, Borehole sealing

## 1. Introduction

Sludge from Bang Khen Water Treatment Plant, Metropolitan Waterworks Authority of Thailand (MWA) has increased up to 247 tons/day. The sludge has been collected from the water treatment process. The sludge volume depends on the amount of sediment transported by rain water in raw water source. The sludge cake is normally used to fill in abandoned land. After several years, it results in an excessively high deposition. Utilization of the sludge for other purposes is being considered in order to reduce the volume of the sludge [1].

One of solutions is to use the sludge to minimize brine circulation in a rock salt formation. A common solution practice in the construction industry is to use bentonite-mixed with cement as a grouting material to reduce permeability of voids and openings in the rock salt. The lack of a true understanding of the permeability characteristics of the sludge-mixed cement makes it difficult to predict the brine flow in geological repository under complex hydrogeological environments [2-3].

## 2. Grouts Preparation

The sludge-mixed cement mixing is performed according to API NO.10 [4-6] by mixing cement with sludge (particle size less than 0.0075 mm) and salt (NaCl) saturated brine. The components of sludge-mixed cement slurry are commercial grade Portland cement mixed with chloride resistant agent (Portland-pozzolan cement, type IP), sludge, NaCl saturated brine and anti-form agent. The brine is prepared by dissolving clean rock salt in distilled water. Anti-form agent is used to decrease the air content of the cement of the cement sludge and therefore, it is easy to control the cement slurry weight and volume. Table 1 gives the weight composition of mixture. Sludge is tested to determine the Atterberg's limits, specific gravity, and particle size distribution. The equipment and test procedure follow the ASTM standards (D422, D854) [7-8]. The average liquid limit, plastic limit and plasticity index are 55%, 22% and 23%, respectively. The average specific gravity is 2.56. Figure 1 shows the particle size distributions of the sludge used here [1]. All grouts are prepared by mixing at the brine-to-sludge-mixed cement ratio of 1:1. The saturated brine is poured into the mixing container at a low mixture 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 slurry mixtures are poured and cured in 54 mm diameter PVC mold for the mechanical characterization test. Molds are cured under atmospheric pressure at room temperature (28 to 30°C). Over 240 specimens are prepared for testing.

## 3. Basic Mechanical Characterization of Grouting Materials

The basic mechanical properties of the mixtures are determined to select the appropriate proportions of sludge-to-cement ratios. The sludge-mixed cement ratios (S:C) 0:10, 1:10, 2:10, 3:10, 4:10 and 5:10 by weight are prepared with brine-sludge-mixed cement ratio of 1:1. Characterization testing provides the uniaxial compressive strength ( $\sigma_c$ ), Young's modulus (E), Brazilian tensile strength ( $\sigma_B$ ), and the viscosity and slurry density of cement grout. The 54 mm diameter cylindrical sludge-mixed cement specimens with length to diameter ratios between 2.5 and 3.0 are prepared by curing cement pastes in PVC molds for 7, 14, 21 and 28 days. They are cured at room temperature (ASTM C192) [9]. 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.

A compression machine, SBEL model PLT-75 is used to test the specimens. The uniaxial compressive strength test procedure follows, as much as practical, the ASTM standards (D7012 and C39) [10-11]. During test, the axial and lateral deformations are monitored. The failure load is recorded. All of the specimens experienced splitting failure. Figure

2 and 3 show the average compressive strengths and elastic modulus as function of curing time.

The Brazilian tensile strength tests are performed in accordance with ASTM standard (D3967) [12]. The load is applied along the diameter of each specimen until failure occurred. Figure 4 shows the average Brazilian strengths as function of curing time.

Viscosity measurement follows, as much as practical, ASTM D2196 [13]. Table 2 shows results of six viscosity and density measurements with sludge–mixed cement ratios (S:C) 0:10, 1:10, 2:10, 3:10, 4:10 and 5:10, respectively. The viscosity is measured with Brookfield® viscometer. The dynamics viscosity of cement slurries tends to increase as the sludge–mixed cement ratios increase.

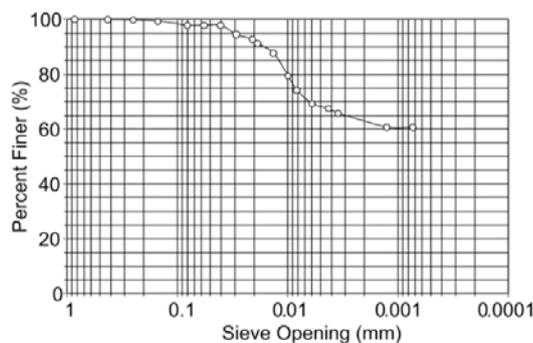
#### 4. Rock Salt Specimens

Fifty–four mm diameter core samples of the rock salt are donated by Pimai Salt Company Limited, Nakhon Ratchasima province. They are collected from a borehole located in Pimai district. All samples are obtained from the Middle Salt members at depths between 30 m and 70 m of Khorat basin. Sample preparation are followed the ASTM D4543 standard practice [14], as much as practical. Six specimens are prepared for the push–out resistance test with length about 100 mm. After preparation, the specimens are labeled and wrapped with plastic film. The specimen designation is identified.

The petrographic properties of the tested cores are as follows. The salt crystals are tightly interlocked. The diameters of milky white halite range from 0.1 to 0.2 cm. Smoky dark halite is associated with anhydrite and clay inclusions. The associated minerals include local occurrence of carnallite. Some large crystals ranging from 2.0 to 3.0 cm are locally occurred [15].

**Table 1** Weight composition of sludge–mixed cement slurry.

Materials	Weight (g)
Portland cement (type V) mixed with sludge	700
NaCl saturated brine	700
Anti–form agent	20
Liquid additive	3.5



**Figure 1** Grain size distribution of water treatment sludge [1].

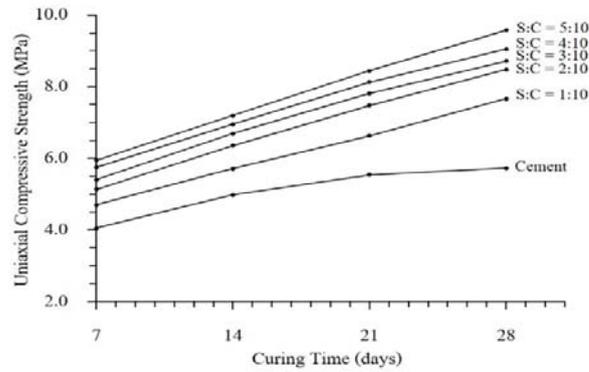


Figure 2 Uniaxial compressive strength of sludge-mixed cement as a function of curing time.

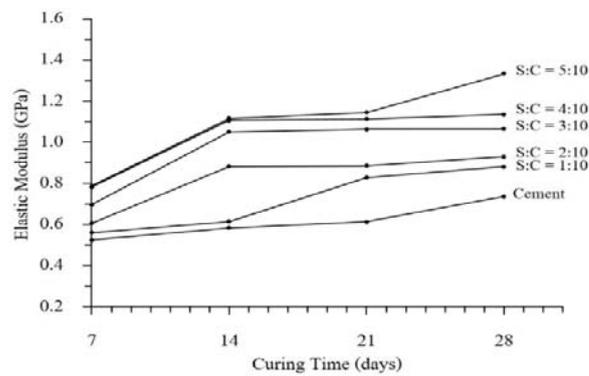


Figure 3 Elastic modulus of sludge-mixed cement as a function of curing time.

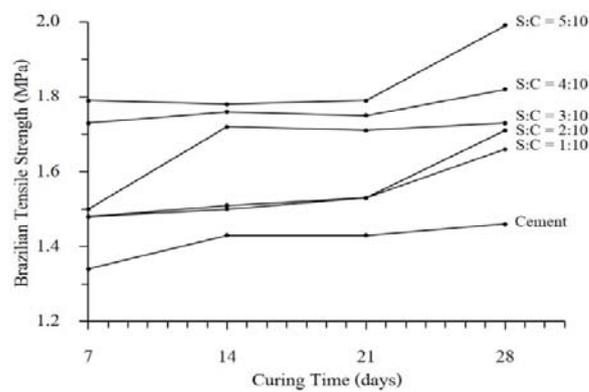


Figure 4 Brazilian tensile strength of sludge-mixed cement as a function of curing time.

**Table 2** Viscosity and slurry density of cement slurry, performed at room temperature and atmospheric pressure following standard ASTM D2196 [13].

Type	Mix ratio	Temperature (°C)		Cement Slurry Density (g/cc)	Dynamic Viscosity ( $\times 10^3$ mPa·s)
		Room	Slurry		
C	0:10	30.1	27.4	1.56	1.27
S:C	1:10	31.0	28.0	1.65	1.07
S:C	2:10	30.2	28.0	1.71	1.33
S:C	3:10	30.4	28.5	1.72	1.53
S:C	4:10	29.5	27.5	1.73	1.59
S:C	5:10	30.2	27.8	1.75	2.28

### 5. Push-out Resistance Test

The objective of this test is to determine the axial mechanical strength of rock salt core plugs in borehole through push-out tests. The curing period for all push-out tests is 7 days. Figure 5 shows the schematic drawing of push-out test setup. An axial load is applied to a rock salt core plug through an axial bar and cylindrical steel rod. The top displacement of the borehole plug is measured by dial gages. The axial load is measured by a load gage of hydraulic pump. A cylindrical steel plate is centered on the bottom bearing plate of the axial bar. A square platen with a central hole is centered on the circular steel plate with a slit. All of 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. Figure 6 shows the push-out test setup. A sludge-mixed cement cylinder in PVC molds with rock salt core is centered on the circular platen. The specimens are loaded under constant stress. The load and top plug displacements are recorded manually at 10 seconds intervals until the sample failed.

The bond strength (the average shear stress,  $\tau_{av}$ ) distribution induced by push-out test loading along the rock salt/cement plug interface can be calculated by the following equation [16]:

$$\tau_{av} = F/\pi D_i L \quad (1)$$

where F is the failure load,  $D_i$  is the plug diameter and L is the rock salt plug length. The dimensions of the sludge-mixed cement cylinders and the bond strength of rock salt core plugs are summarized in Table 3.

Figure 7 plots the applied axial stress as a function of the top plug displacements. The peak and residual shear stress are plotted against the corresponding S:C ratios. Figure 8 shows the post-test specimens after failure in the push-out test. Rock bridges fail at applied axial stresses from 17.77 to 118.01 MPa. The bond strength tends to increase as the sludge-mixed cement ratios (S:C) increase. The highest bond strength is observed from the ratio of S: B = 5:10. Figure 9 shows sample which is cut along the axis after failure. The thick rock salt residue on the sludge-mixed cement borehole walls above the (slipped) rock salt core plug and absence of dissolution indicate a good bonding.

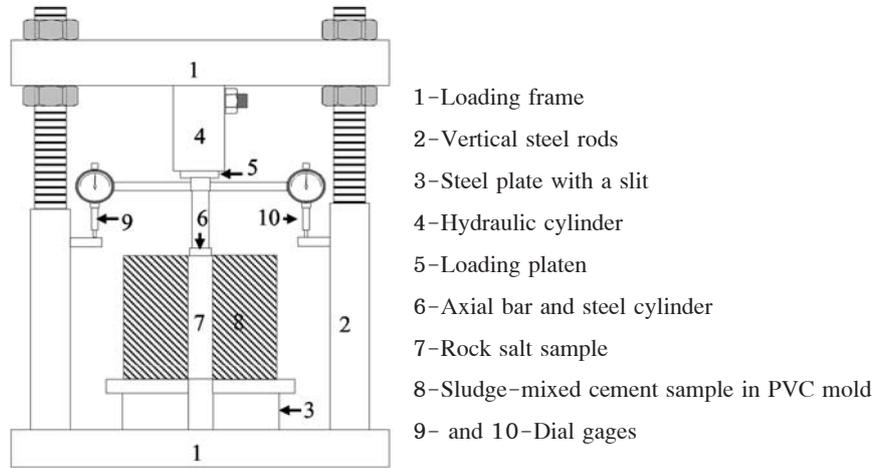


Figure 5 Schematic drawing of push-out resistance test setup.



Figure 6 Push-out test setup.

Table 3 Dimensions of cement cylinders used for push-out tests, the axial strength ( $\sigma_{ax}$ ) and average shear strength ( $t_{av}$ ).

Type	Mix ratio	$D_o$ (mm)	$L_o$ (mm)	$D_i$ (mm)	L (mm)	F (kN)	$\sigma_{ax}$ (MPa)	$t_{av}$ (MPa)
C	0:10	100.30	101.20	25.40	101.20	9.0	17.77	1.12
S:C	1:10	100.68	101.30	25.40	100.30	24.0	47.39	3.00
S:C	2:10	100.68	101.65	25.30	100.65	25.0	49.75	3.13
S:C	3:10	100.60	101.30	25.40	100.30	40.0	78.98	5.00
S:C	4:10	100.12	101.20	25.60	100.02	46.0	89.41	5.72
S:C	5:10	100.36	101.21	25.45	100.21	60.0	118.01	7.49

$D_o$  = cement cylinder diameter,  $L_o$  = cylinder length,  $D_i$  = plug diameter, L = rock salt plug length, F = axial load at failure

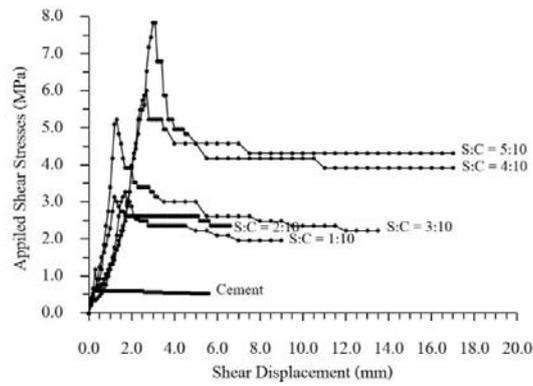


Figure 7 Applied shear stress vs. top axial displacement for the push-out resistance test.

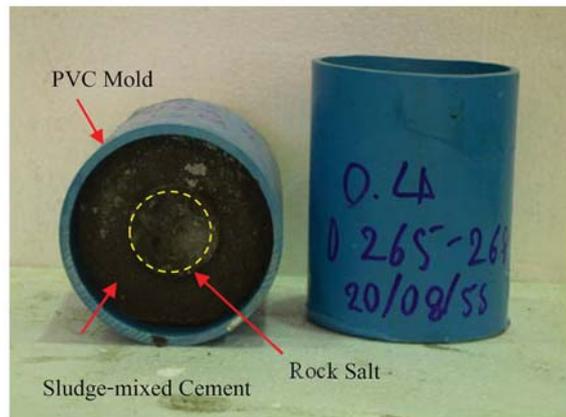


Figure 8 Example of specimens after failure in the push-out test.

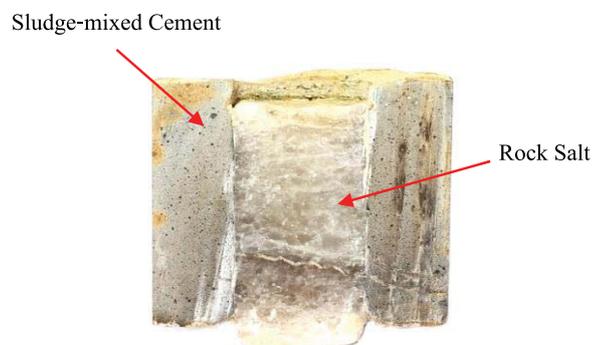


Figure 9 A cut section of a specimen after failure in the push-out test.

## 6. Permeability Testing of Sludge-mixed Cement Grout

The permeability of grouting materials is determined in terms 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 the system considered herein is measured using a constant head apparatus as shown in Figure 10. The flow in longitudinal direction of a tested system is described by Darcy's law. The coefficient of permeability,  $K$ , can be calculated from the equation [17]:

$$K = Q/Ai \quad (2)$$

where  $Q$  is volume flow rate ( $m^3/s$ );  $A$  is cross-sectional area of sludge-mixed cement grout ( $m^2$ ); and  $i$  is the hydraulic gradient. The intrinsic permeability ( $k$ ) can be determined from the equation:

$$k = K\mu/\gamma_w \quad (3)$$

where  $K$  is the coefficient of permeability ( $m/s$ );  $\mu$  is dynamic viscosity of saturated brine and  $\gamma_w$  is density of liquid water at 20 degree Celsius ( $9,789 N/m^3$ ). The cylinder specimen is 10 cm in diameter and 10 cm long. The permeability of the test system is measured and recorded at 7, 14, and 28 days of curing period. 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 sludge-mixed cement grouts as a function of curing time are shown in Figures 11 and 12, respectively.

## 7. Discussions, Conclusions, and Recommendations

The sludge-mixed cement grouts are prepared from the commercial grade Portland cement mixed with Bang Khen water treatment sludge, saturated brine and chloride resistant agent have been tested to determine the mechanical and hydraulic performance. This study aims to determine the appropriate slurry viscosity and strength of the sludge-mixed cement grouting. The results lead to the selection of the most suitable ratio of sludge-mixed cement (S:C) for grouting in rock salt.

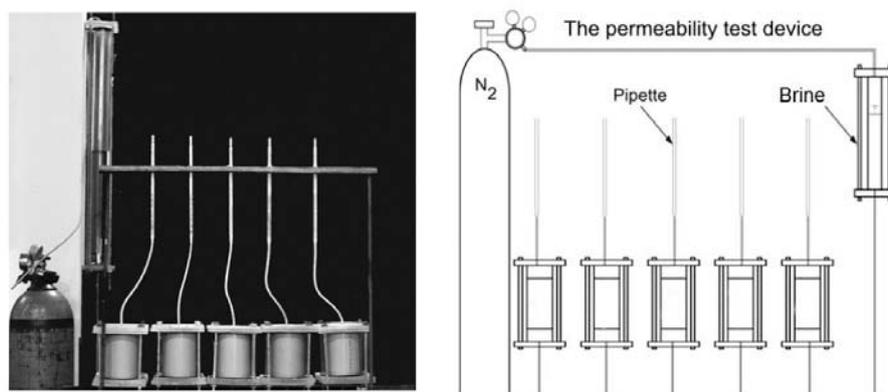


Figure 10 Constant head flow test apparatus used for permeability testing of grouting materials.

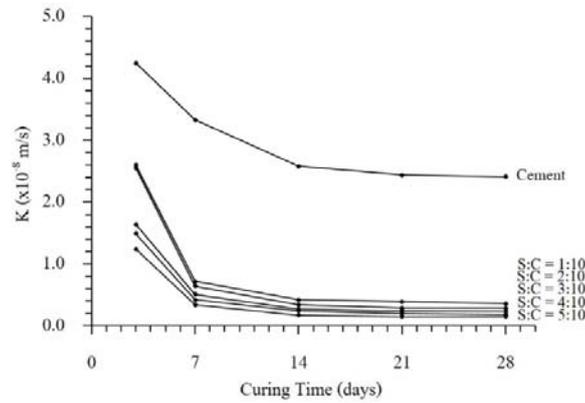


Figure 11 Conductivity of permeability of sludge-mixed cement as a function of curing time.

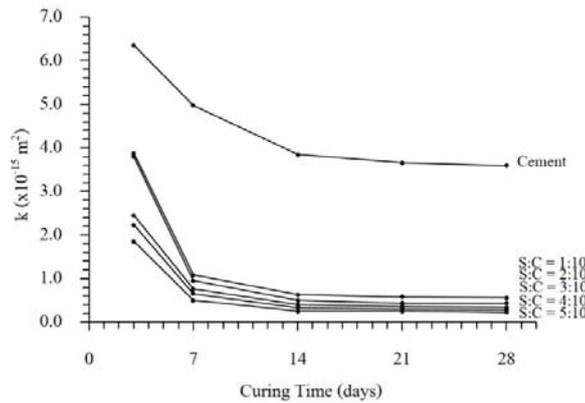


Figure 12 Intrinsic permeability of sludge-mixed cement as a function of curing time.

Six rock salt cores are plugged within cement cylinder for the push-out tests. Average interface shear strengths range from 1.12 to 7.49 MPa with various S:C ratios. Samples showing high axial strength generally lead to high bond strength. The bottom plug displacements are smaller compared to the top axial displacements prior to bond failure. Upon plug slip, the difference between the top and bottom plug displacements mostly decreases, most probably due to stress relief caused by slip along the interface. The Brazilian tensile strength indicates that the bond strength between the cured sludge-mixed cement grout and rock salt varies from 1.34 to 1.82 MPa with a various of S:C ratio.

The permeability of the sludge-mixed cement grouting materials measured from the longitudinal flow test with constant head decreases with curing time at 7, 14, 21 and 28 days. The results indicate that when the curing time increases the intrinsic permeability (k) of cement grout decreases. The mixture with the S:C of 5:10 by weight gives the lowest permeability. The S:C mixtures have the mechanical and hydraulic properties equivalent to those of the commercial grade Portland cement mixtures which indicates that the sludge can be used as a substituted material to mix with cement for rock salt grouting purpose.

This study made no attempt to predict the relation between bond strength of sludge-mixed cement sealing and curing period. The Push-out tests should be performed on plugs with a variety of relatively long curing times.

#### Acknowledgements

This research is supported by Suranaree University of Technology and by Higher Education Research Promotion and National Research University of Thailand, Office of the Higher Education Commission. Permission to publish this paper is gratefully acknowledged. We would like to thank Metropolitan Waterworks Authority of Thailand for donating water treatment sludge and Pimai Salt Company for donating rock salt cores used in this study.

#### References

- [1] K. Wetchasat and K. Fuenkajorn, "Mechanical and Hydraulic Performance of Sludge Mixed Cement Grout in Rock Fractures", 7th Asian Rock Mechanics Symposium, Seoul, Korea, 15-19 October 2012, pp. 1477-1485.
- [2] Fuenkajorn, K., Daemen, J.J.K., Sealing of Boreholes and Underground Excavations in Rock, Chapman and Hall, London, 1996.
- [3] H. Akgün and J.J.K. Daemen, "Analytical and Experimental Assessment of Mechanical Borehole Sealing Performance in rock", Engineering Geology Journal, Vol. 47, 1997, pp. 233-241.
- [4] H. Akgün and J.J.K. Daemen, 1989, Bond Strength of Cement Borehole Plugs in Salt, Tech. Rep. NUREG/CR-5401, U.S. Nuclear Regulatory Commission, Washington, DC, 1989, p. 230.
- [5] H. Akgün and J.J.K. Daemen, "Influence of degree of Saturation on the Borehole Sealing Performance of an Expansive Cement Grout", Cement and Concrete Research Journal, Vol. 30, 2000, pp. 281-289.
- [6] American Petroleum Institute, Specifications for Materials and Testing for Well Cements, 3rd Ed., American Petroleum Institute, Production Department, Dallas, TX., 1986.
- [7] ASTM D422, "Standard Test Method for Particle-Size Analysis of Soils", Annual Book of ASTM Standards 04.08, American Society for Testing and Materials, Philadelphia, 2007.
- [8] ASTM D854, "Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer", Annual Book of ASTM Standards 04.08, American Society for Testing and Materials, Philadelphia, 2010.
- [9] ASTM C192, "Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory", Annual Book of ASTM Standards 04.02, American Society for Testing and Materials, Philadelphia, 2007.
- [10] ASTM D7012, "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 Standard 04.09, American Society for Testing and Materials, Philadelphia, 2010.
- [11] ASTM C39, "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens", Annual Book of ASTM Standards 04.01, American Society for Testing and Materials, Philadelphia, 2010.
- [12] ASTM D3967, "Standard Test Method for Splitting Tensile Strength of Intact Rock Core Specimens, Annual Book of ASTM Standards 04.08, American Society for Testing and Materials, Philadelphia, 1981.
- [13] ASTM D2196, "Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer", Annual Book of ASTM Standard, 06.01, American Society for Testing and Materials, Philadelphia, 2010.
- [14] ASTM D4543, "Standard Test Method for Preparing Rock Core Specimens and Determining Dimensional and Shape Tolerances", Annual Book of ASTM Standard 04.08, American Society for Testing and Materials, Philadelphia, 1985.
- [15] P. Tepnarong, "Bond Strength of Cement Sealing in Maha Sarakham Salt", 7th Asian Rock Mechanics Symposium, Seoul, Korea, 15-19 October 2012, pp. 584-593.
- [16] J.C. Stormont and J.J.K. Daemen, "Axial Strength of Cement Borehole Plugs in Granite and Basalt", Tech. Rep. NUREG/CR - 3594, U.S. Nuclear Regulatory Commission, by the Department of Mining and Geological Engineering, University of Arizona, Tucson, 1983.
- [17] B. Indraratna and P. Ranjith, Hydromechanical Aspects and Unsaturated Flow in Joints Rock, Lisse: A. A. Balkema, 2001.