



MECHANICAL PERFORMANCE OF CONSOLIDATED CRUSHED SALT MIXED WITH $MgCl_2$ BRINE FOR CARNALITE MINE BACKFILL

Worawat Suwannabut¹, Thanittha Thonggrapha² and Kittitep Fuenkajorn^{3*}

¹Student, Geomechanics Research Unit, Suranaree University of Technology, Thailand

²Instructor, Geomechanics Research Unit, Suranaree University of Technology, Thailand

³Professor, Geomechanics Research Unit, Suranaree University of Technology, Thailand

*Corresponding author: kittitep@sut.ac.th

บทคัดย่อ

การศึกษานี้เกี่ยวกับการหาคูมสมบัติเชิงกลศาสตร์ของเกลือหินบดผสมกับแมกนีเซียมคลอไรด์ โดยใช้อัตราส่วน 5 เปอร์เซ็นต์โดยน้ำหนักและภายใต้การอัดตัวคายน้ำที่ระยะเวลาและความเค้นในระดับต่างกัน เม็ดเกลือหินบดมีขนาด 0.075-4.76 มิลลิเมตร ระยะเวลาในการอัดตัวอยู่ระหว่าง 7-90 วัน การอัดตัวคายน้ำอยู่ภายใต้ความเค้นกครระหว่าง 2.5-10 เมกะปาสกาล ผลการตรวจวัดระบุว่าความเครียดที่เกิดขึ้นระหว่างการอัดตัวเพิ่มขึ้นอย่างรวดเร็วในช่วง 7 วันแรกและมีแนวโน้มเข้าสู่ค่าคงที่หลังจาก 30 วัน ความหนาแน่น กำลังกดสูงสุดและสัมประสิทธิ์ความยืดหยุ่นมีค่าเพิ่มขึ้นตามความเค้นและระยะเวลาการอัดตัว แต่ค่าอัตราส่วนปัวซองลดลงอย่างช้า ๆ ตามระยะเวลา กลไกหลักที่ทำให้เกลือหินบดมีความแข็งและความแกร่งเพิ่มขึ้นคือ การเปลี่ยนแปลงปริมาตร โดยรวมอันเนื่องมาจากการจัดเรียงตัว การแตกและการเชื่อมประสานระหว่างเม็ดเกลือหินบด พลังงานความเครียดเฉลี่ยของตัวอย่างภายใต้การบดอัดถูกคำนวณขึ้นเพื่อใช้ในการคาดคะเนคูมสมบัติเชิงกลศาสตร์ของเกลือบดอัดที่ใช้เป็นวัสดุถมกลับในช่องเหมืองคาร์นาลไลต์

ABSTRACT

Mechanical properties of consolidated crushed salt mixed with 5% $MgCl_2$ brine by weight are determined under different stresses and periods. The crushed salt specimens with particle sizes ranging from 0.075 to 4.76 mm are consolidated under stresses ranging from 2.5 to 10 MPa for 7 to 90 days. The consolidations strains increase rapidly within the first 7 days and tend to approach a limit value after 30 days. The density, uniaxial compressive strength and elastic modulus measured after consolidation increase with the applied stress and duration. The Poisson's ratio decreases slowly with time. The mechanisms that strengthen and stiffen the crushed salt mass are consolidation by volumetric change due to particle rearrangement and cracking, and healing between crushed salt particles. The mean strain energy required during consolidation for each specimen is calculated. The relations can be used to predict the crushed salt performance installed in potash (carnallite) mine openings.

KEYWORDS: Crushed salt, $MgCl_2$ Brine, Compressive Strength, Elastic Modulus, Strain Energy

1. Introduction

Salt tailing has long been considered as sealing material in abandoned openings in salt and potash mines due to its availability, low cost and physical, chemical and mechanical compatibility with the host rock [1, 2]. The understanding of the consolidation behavior of crushed salt is thus the primary concern for the long-term assessment of the performance of the seal. The factors affecting the mechanical performance of consolidated crushed salt are moisture, duration, consolidation stresses, initial density, particle size and temperature [3-6]. Case et al. [7] conclude from their experimental work that the volumetric creep strain rate increases with time and does not reach steady state values even after 1 to 2 months of load application. The uniaxial compressive strength and Young's modulus of crushed rock salt also increase with densification time [8-10]. Even though the effects of consolidation on the physical and hydraulic behavior of crushed salt have been recognized and studied, experimental determination of the mechanical properties of crushed salt after consolidation has rarely been investigated. Most of the investigations on crushed salt behavior under loading mentioned earlier have used saturated NaCl brine as mixing fluid to prepare specimens for compaction and consolidation testing. A recent experimental study by Theerapun et al. [11] suggest however that the presence of NaCl brine as mixing fluid in backfill can dissolve the surrounding carnallite in potash mine opening, and hence reduces the mechanical performance of support pillars and mine roof and floor. The carnallite and halite are however insensitive to $MgCl_2$ (magnesium chloride) brine. The $MgCl_2$ brine is a toxic waste product obtained from the processing of carnallite ore excavated in Thailand. Disposal of the $MgCl_2$ brine and crushed salt by returning them to the abandoned mine openings not only minimizes the long-term surface subsidence, but also prevents the environmental contamination from these materials.

The objective of this study is to predict the physical and mechanical properties of crushed salt backfill after installed in carnallite mine opening. Consolidation tests are performed on crushed salt mixed with saturated $MgCl_2$ brine under axial stresses of 2.5, 5, 7.5 and 10 MPa for 7 to 90 days. The specimen properties are determined as a function of mean strain energy densities applied during consolidation. To demonstrate the strain energy approach, time-dependent closure of circular openings is calculated to determine the released mean strain energy density as a function of time after excavation. The backfill properties under the released mean strain energy can be predicted for different opening depths and installation periods.

2. Crushed Salt

The crushed salt specimen is prepared from salt blocks obtained from the Lower member of Maha Sarakham formation in Khorat basin. The salt blocks are crushed by hammer mill to produce particle sizes ranging from 0.075 to 4.75 mm (Figure 1). This size range is equivalent to those expected to be obtained as waste product from the local potash mines. The sphericity and roughness are determined from individual particles using an optical microscope. Based on the Power [12] classification systems the crushed salt is classified as angular to sub-angular with spherical shape.

Saturated $MgCl_2$ brine is prepared by mixing $MgCl_2$ tablets with distilled water. The proportion of $MgCl_2$ to water is about 57% by weight. Specific gravity of the saturated $MgCl_2$ brine ($S.G._{MgCl_2,B}$) is calculated by: $S.G._{MgCl_2,B} = \rho_{MgCl_2,B} / \rho_{H_2O}$, where $\rho_{MgCl_2,B}$

is density of saturated $MgCl_2$ brine (measured by a hydrometer in kg/m^3) and ρ_{H_2O} is density of water. The specific gravity of the saturated $MgCl_2$ brine used here is 1.30 at $21^\circ C$.

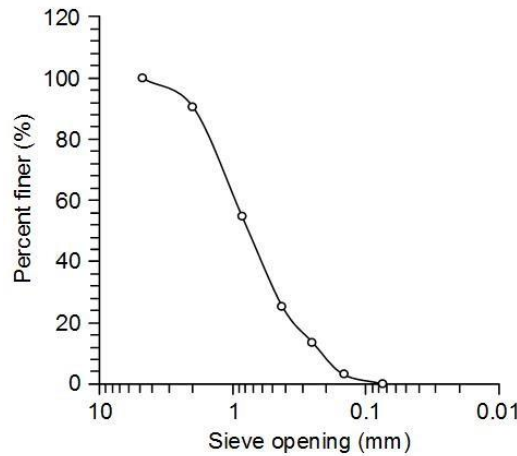


Figure 1 Grain size distribution of crushed salt

3. Test Apparatus and Method

A thick-wall stainless steel tube with inner diameter of 54 mm and outer diameter of 64 mm is used as consolidation chamber (Figure 2). Two loading pistons fitted with rubber O-rings are used to apply axial stress on the opposite ends of the specimen. The piston has a drainage hole to allow excess fluid to flow out during consolidation. A consolidation load frame applies constant stresses ranging from 2.5, 5, 7.5 to 10 MPa. The axial displacements are monitored by dial gages and are used to calculate the axial (consolidation) strains. The applied load is removed after 7, 15, 30 and 90 days. The initial density and porosity before loading for all specimens are measured. The specimen is then carefully pushed out from the stainless-steel tube and prepared for the mechanical testing. A total of 16 specimens have been consolidated under different stresses and periods.

The suitable $MgCl_2$ brine content is first determined by applying consolidation stresses from 2.5, 5, 7.5 to 10 MPa to the crushed salt mixed with saturated $MgCl_2$ brine for 0, 5 and 10% by weight. After the crushed salt and $MgCl_2$ brine are mixed thoroughly, they are poured into the steel tube, and lightly tapped to obtain a flat end. The constant consolidation stress (σ_{cons}) is then applied. The results indicate that axial strain increases with applied stress and $MgCl_2$ brine content (Figure 3). The dry specimen shows the lowest consolidation. The axial strains curves obtained at 5% and 10% brine contents are similar, suggesting that increasing the $MgCl_2$ brine content beyond 5% does not enhance the consolidation rates. The saturated $MgCl_2$ brine content used in this study is therefore maintained constant at 5% by weight.

4. Consolidation Test Results

The axial strains (ϵ_{cons}) are plotted as a function of consolidation period (t) up to 90 days (Figure 4). A higher applied stress (σ_{cons}) induces a larger strain. The strains increase rapidly within 7 days after the load application. The strain rates decrease with time, and tend to remain relatively constant after 30 days. The deformation of the specimens can be divided into two phases: instantaneous and transient

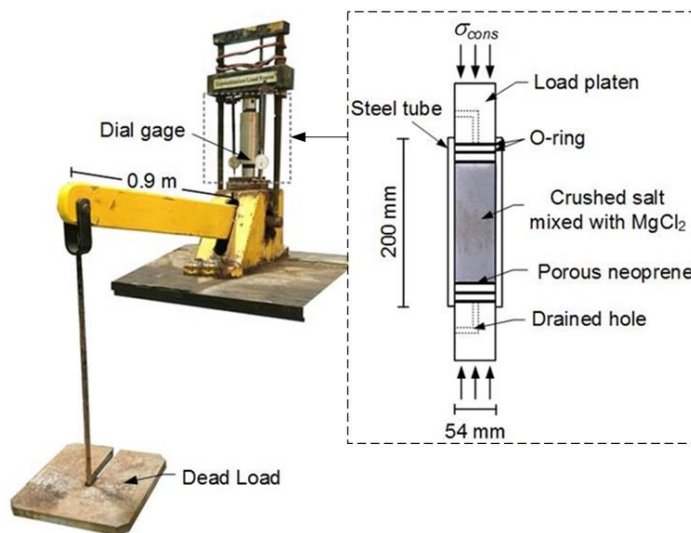


Figure 2 Consolidation apparatus and consolidation steel tube

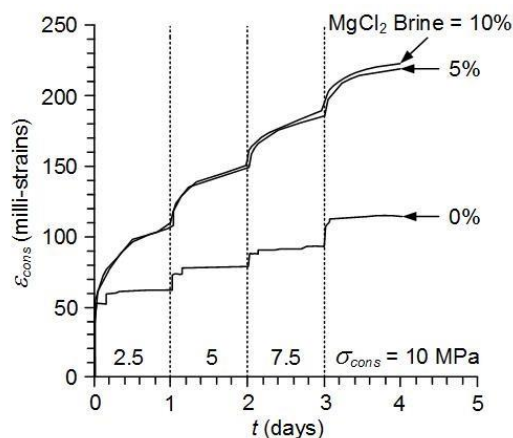


Figure 3 Axial strains as a function of time for different consolidation stresses

deformations. The mechanism governing the instantaneous deformation is probably the rearrangement of the salt particles which occurs immediately after load application. The transient phase may involve cracking and creep deformation of the salt particles.

5. Mechanical Properties of Consolidated Crushed Salt

Uniaxial compression tests are performed on the crushed salt specimens after removing from the consolidation tube. The specimens appear solidified, particularly under long consolidation period and high stress. They are cut to obtain flat and parallel end surfaces. The length-to-diameter ratios are about 2.0-2.2 which comply with the specifications given by the ASTM standard

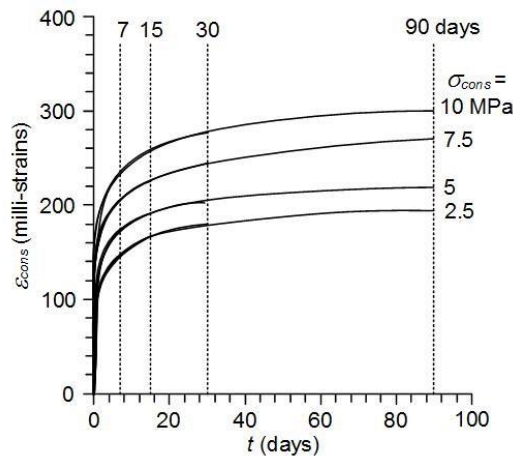


Figure 4 Axial strain (ϵ_{cons}) as a function of consolidation period (t) for different consolidation stresses (σ_{cons})

practice [13]. The specimen density is determined. The axial stress is applied by a compression load frame. The axial and lateral deformations are monitored. The uniaxial compressive strengths and elastic parameters are determined in accordance with the ASTM standard practice [14]. The stress-strain curves obtained from different consolidation stresses and periods are plotted in Figure 5. For all σ_{cons} 's, higher consolidation period gives higher strengths. Specimens under higher consolidation stresses and longer periods tend to be more solidified than those under lower ones. The elastic parameters are calculated as tangent at 50% of the failure stress. Table 1 summarizes the test results. Larger consolidation period and stress lead to higher density, strength and elasticity of the specimens. This effect becomes larger under higher σ_{cons} . The Poisson's ratios however decrease slightly with increasing consolidation period and stress. The results suggest that the crushed salt becomes denser, stiffer, stronger and less compressible with increasing consolidation stress and duration. This agrees reasonably well with the results obtained from Wang et al. [8] and Miao et al. [9] who report that under consolidation stress of 15 MPa the strength and elastic modulus of crushed salt can increase up to 28.4 MPa and 9.64 GPa after being consolidated for 97 days.

Table 2 compares the mechanical properties of crushed salt mixed with $MgCl_2$ brine obtained here with the same crushed salt but mixed with NaCl brine tested by Khamrat et al. [10]. The comparisons are made for the same consolidation stress ($\sigma_{cons} = 10$ MPa) and period (90 days). The strength and elastic modulus of the crushed salt NaCl mixture are significantly higher than those of the crushed salt $MgCl_2$ mixture. This is primarily because the NaCl brine can recrystallize easier and quicker than the $MgCl_2$ brine. The recrystallization process of NaCl brine can increase the strength and stiffness of the consolidated crushed salt.

The difference of the recrystallization behaviors between the two solutions is supported by the experimental evidence obtained by Sander and Herbert [15].

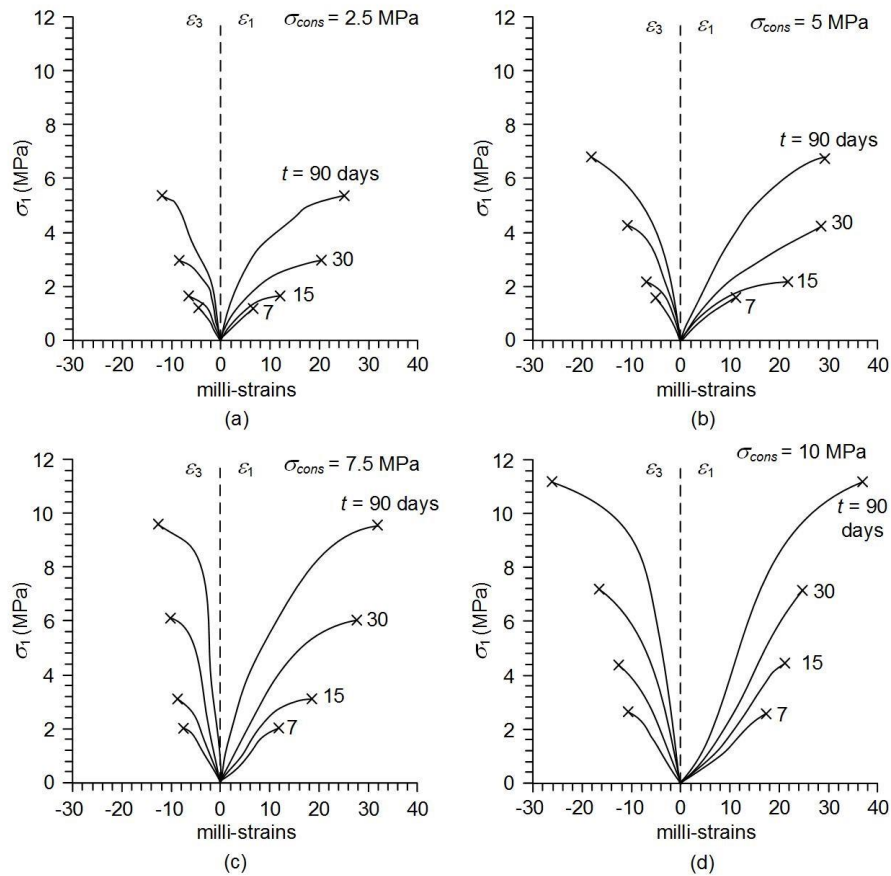


Figure 5 Stress–strain curves obtained from uniaxial compression test of crushed salt specimens after consolidation

6. Strain Energy Density Principle

The strain energy density required to consolidate the crushed salt specimens under different stresses and periods is calculated from the test results. The mean strain energy (W_m) can be calculated from the applied mean stresses (σ_m) and strains (ϵ_m):

$$W_m = \frac{3}{2}(\sigma_m \cdot \epsilon_m) \tag{1}$$

$$\sigma_m = \frac{1}{3}(\sigma_1 + \sigma_2 + \sigma_3) \tag{2}$$

$$\epsilon_m = \frac{1}{3}(\epsilon_1 + \epsilon_2 + \epsilon_3) \tag{3}$$

Table 1 Mechanical properties of crushed salt after consolidation.

σ_{cons} (MPa)	Parameters	t (days)			
		7	15	30	90
2.5	ρ (g/cm ³)	1.52	1.54	1.58	1.62
	σ_c (MPa)	1.12	1.62	2.97	5.32
	E (MPa)	150	192	224	269
	ν	0.36	0.35	0.34	0.33
5	ρ (g/cm ³)	1.56	1.61	1.63	1.67
	σ_c (MPa)	1.62	2.34	4.22	6.72
	E (MPa)	179	213	261	303
	ν	0.35	0.34	0.33	0.32
7.5	ρ (g/cm ³)	1.64	1.68	1.70	1.76
	σ_c (MPa)	2.03	3.12	5.94	9.51
	E (MPa)	210	247	290	330
	ν	0.34	0.33	0.32	0.31
10	ρ (g/cm ³)	1.68	1.75	1.78	1.86
	σ_c (MPa)	2.66	4.37	7.03	11.67
	E (MPa)	246	271	314	377
	ν	0.33	0.32	0.31	0.30

Table 2 Mechanical properties of crushed salt after consolidation under $\sigma_{\text{cons}} = 10$ MPa for 90 days comparing between crushed salt mixed with MgCl₂ brine (tested here) and with NaCl brine obtained from Khamrat et al. [10]

Mixing Fluid	Parameters			
	ρ (g/cm ³)	σ_c (MPa)	E (MPa)	ν
NaCl brine	1.58	15.07	439	0.29
MgCl ₂ brine	1.86	7.65	377	0.30

In the consolidation tube, the lateral stresses σ_2 and σ_3 on the specimen are equal and can be calculated as a function of time based on the uniaxial strain condition, i.e. assume that no lateral strain occurs in the thick-wall steel tube during consolidation. The axial strains from the measurement results, therefore, represent the volumetric strain. The lateral stresses can be calculated by:

$$\sigma_2 = \sigma_3 = \left[\frac{\nu}{(1-\nu)} \right] \sigma_1 \quad (4)$$

where ν is Poisson's ratio, and σ_1 is consolidation stress (σ_{cons}). Table 3 gives the calculated mean stress and strain and strain energy density for different consolidation stresses and durations. The specimens under higher consolidation stress and time are obtained under higher mean strain energy densities.

The changes of physical and mechanical properties of crushed salt can be related with the applied mean strain energy density during consolidation. Two main mechanisms govern the changes of the properties of crushed salt: consolidation and healing [16]. The first mechanism involves the volumetric reduction by particle rearrangement, creep, cracking and sliding between grain boundaries. It is reflected by instantaneous and transient deformations which are mainly controlled by the applied energy. The second mechanism involves the healing between salt particles. This mechanism does not decrease the crushed salt volume. It can however strengthen and stiffen the specimen as the consolidation period increases.

Table 3 Mean stresses, strains and strain energy densities applied to crushed salt during consolidation

σ_{cons} (MPa)	Parameters	t (days)			
		7	15	30	90
2.5	σ_m (MPa)	1.79	1.74	1.71	1.67
	ϵ_m (10^{-3})	49.1	54.0	60.3	67.3
	w_m (kPa)	132	141	154	168
5	σ_m (MPa)	3.45	3.33	3.31	3.23
	ϵ_m (10^{-3})	61.3	64.5	67.7	72.2
	w_m (kPa)	318	329	336	350
7.5	σ_m (MPa)	5.09	4.91	4.87	4.74
	ϵ_m (10^{-3})	68.2	74.4	81.5	88.5
	w_m (kPa)	520	459	595	630
10	σ_m (MPa)	6.61	6.47	6.33	6.05
	ϵ_m (10^{-3})	72.2	86.8	92.4	100.2
	w_m (kPa)	769	842	877	910

Based on the concept above, the crushed salt density (ρ) can be represented by:

$$\rho = \rho_{initial} + \Delta\rho_{cons} \tag{5}$$

where $\rho_{initial}$ is the initial density before applying the mean strain energy (equal to 1.30 g/cm^3) and $\Delta\rho_{cons}$ is the reduction of bulk density due to the strain energy and consolidation period. Regression analysis of the test data by SPSS software can determine $\Delta\rho_{cons}$ as a function of w_m and t :

$$\Delta\rho_{cons} = 0.345W_m^{0.353}t^{0.109} \text{ (g/cm}^3\text{)} \quad (6)$$

Good correlation is obtained ($R^2 > 0.9$). The curve fits and the test results are compared in Figure 6a. It should be noted that the crushed salt density is independent of the healing mechanism because this mechanism does not reduce the bulk volume of specimens.

Similar to the density prediction above, the reduction of crushed salt porosity (n) during consolidation can be determined by the regression analysis of the test data:

$$n = n_{initial} - \Delta n_{cons} \quad (7)$$

where $n_{initial}$ is the initial crushed salt porosity (39.79%) and Δn_{cons} is the porosity reduction which can be represented by a power equation:

$$\Delta n_{cons} = 16.32W_m^{0.340}t^{0.104} \text{ (%) } \quad (8)$$

The diagrams in Figure 6b suggest that the applied mean strain energy affects the porosity reduction and the density increase more than does the consolidation period.

The crushed salt strength (σ_c) and elasticity (E) are controlled by consolidation energy and period, which can be determined from the test data (Figures 6c and 6d) as:

$$\sigma_c = 1.29W_m^{0.560}t^{0.490} \text{ (MPa)} \quad (9)$$

$$E = 176W_m^{0.220}t^{0.175} \text{ (MPa)} \quad (10)$$

The relationships between the mechanical properties of the crushed salt and its mean strain energy required during each consolidation period, as shown in Figure 6, can be used to predict the performance of crushed salt backfill under in-situ condition.

7. Discussions and Conclusions

The density, strength and elastic parameters of the crushed salt are tested for different consolidation stresses and durations (Table 1). The empirical equations relating the crushed salt properties with the applied mean strain energy density are derived. The application of the strain energy principle allows considering both stress and strain to which the crushed salt specimens are subjected (Figure 6). Two main mechanisms occur during consolidation: (1) volumetric change due to particle rearrangement and cracking and creep of salt crystals, and (2) healing process. Both can strengthen and stiffen the crushed salt specimens.

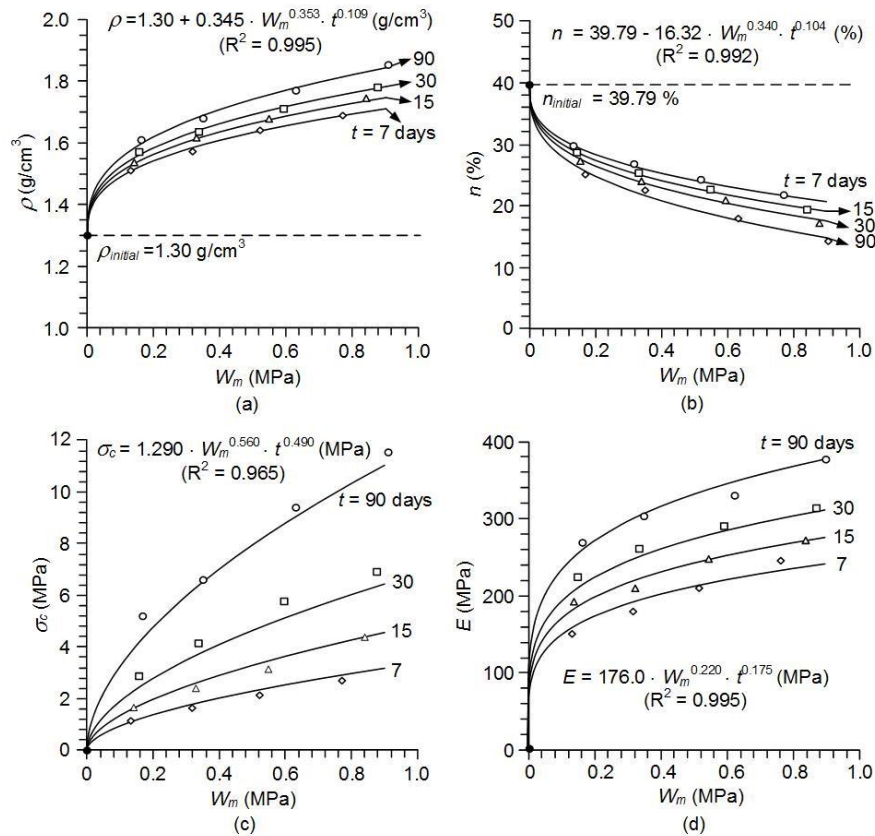


Figure 6 Density (a), porosity (b), uniaxial compressive strength (c) and elastic modulus (d) as a function of mean strain energy density (W_m) applied during consolidation

It is postulated that the time at which the backfill is installed is an important factor governing the mechanical performance of the crushed salt, particularly under great depth. Under shallow depth the crushed salt density and porosity cannot be effectively increased because the available mean strain energy at the opening boundary is low [10].

It should be noted that different grain size distributions and moisture contents of the crushed salt are likely to result in different consolidation behaviors. The properties of the consolidated crushed salt after installation can improve only if it is installed in the openings excavated in time-dependent rocks; such as salt and potash, where creep closure of the opening occurs after excavation. The mechanical properties predictions are also sensitive to the creep parameters of the surrounding rock. Application of different constitutive creep models for the surrounding salt mass may also result in different predictions of the crushed salt properties.

Acknowledgements

This study is funded by Suranaree University of Technology and by the Higher Education Promotion and National Research University of Thailand. Permission to publish this paper is gratefully acknowledged.

References

- [1] Holcomb, D. J. and Hannum, D. W. *Consolidation of crushed-salt backfill under conditions appropriate to the WIPP facility*, Report No. SAND82-0630, Sandia National Labs, Albuquerque. United States, 1982.
- [2] Hansen, F. D. Reconsolidating salt: compaction, constitutive modeling, and physical processes. *International Journal of Rock Mechanics and Mining Sciences*, 1997, 34 (3–4), pp. 119.e1-119.e12.
- [3] Pfeifle, T. W. et al. *Influence of variables on the consolidation and unconfined compressive strength of crushed salt*, Technical report BMI/ONWI-627, prepared by RE/SPEC, Inc. Rapid City. SD. United States, 1987.
- [4] Broome, S.T. et al. Mechanical response and microprocesses of reconsolidating crushed salt at elevated temperature. *Rock Mechanics and Rock Engineering*, 2015, 48 (6), pp. 2615-2629.
- [5] Mills, M. M. et al. Micromechanical processes in consolidated granular salt. *Engineering Geology*, 2018, 239, pp. 206-213.
- [6] Lampe, B. C. et al. Experimental investigation of the influence of pore pressure and porosity on the deformation of granular salt. *International Journal of Rock Mechanics and Mining Sciences*, 2018, 110, pp. 291-305.
- [7] Case, J. B. et al. Laboratory investigation of crushed salt consolidation. In: *Proceedings of the 28th U.S. Symposium on Rock Mechanics (USRMS)*, Tucson. Arizona, 1987.
- [8] Wang, M. L. et al. Deformation mechanisms of WIPP backfill. *Radioactive Waste Management and Environmental Restoration*, 1995, 20 (2-3), pp. 191-211.
- [9] Miao, S. et al. Constitutive models for healing of materials with application to compaction of crushed rock salt. *Journal of Engineering Mechanics*, 1995, 121 (10), pp. 1122-1129.
- [10] Khamrat, S. et al. Crushed salt consolidation for borehole sealing in potash mines. *Geotechnical and Geological Engineering*, 2018, 36 (1), pp. 49-62.
- [11] Theerapun, C. et al. Effects of backfill compositions on the integrity of underground salt and potash mines. *Engineering Journal of Research and Development*, 2017, 28 (2), pp. 15-22.
- [12] Power, M. C. *Comparison charts for estimating roughness and sphericity*. AGI data sheets. American Geological Institute, Alexandria VA, 1982.
- [13] American Society for Testing and Materials. D4543: 1985. *Standard practice for preparing rock core specimens and determining dimensional and shape tolerances: Annual Book of ASTM Standards*, West Conshohocken, PA, 1985.
- [14] American Society for Testing and Materials. D2938: 1995. *Standard test method for unconfined compressive strength of intact rock core specimens: Annual Book of ASTM Standards*, West Conshohocken, P.A, 1995.
- [15] Sander, W. and Herbert, H. J. NaCl crystallization at the MgCl₂/NaCl solution boundary—a possible natural barrier to the transport of radionuclides. *Mineralogical Magazine*, 1985, 49 (351), pp. 256-270.
- [16] Callahan, G.D. et al. *Constitutive behavior of reconsolidating crushed salt*, Rep. No. SAND98-0179, Sandia national laboratories, Albuquerque, NM, 1998.