

# Crushed Salt Reconsolidation at Elevated Temperatures

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**ABSTRACT:** There is a long history of testing crushed salt as backfill for the Waste Isolation Pilot Plant program, but testing was typically done at 100°C or less. Future applications may involve backfilling crushed salt around heat-generating waste packages, where near-field temperatures could reach 250°C or hotter. A series of experiments were conducted to investigate the effects of hydrostatic stress on run-of-mine salt at temperatures up to 250°C and pressures to 20 MPa. The results of these tests were compared with analogous modeling results. By comparing the modeling results at elevated temperatures to the experimental results, the adequacy of the current crushed salt reconsolidation model was evaluated. The model and experimental results both show an increase in the reconsolidation rate with temperature. The current crushed salt model predicts the experimental results well at a temperature of 100°C and matches the overall trends, but over-predicts the temperature dependence of the reconsolidation. Further development of the deformation mechanism activation energies would lead to a better prediction of the temperature dependence by the crushed salt reconsolidation model.

## 1. INTRODUCTION

There is a long history of testing crushed salt backfill for the Waste Isolation Pilot Plant program, but all testing was done at 100°C or less [1]. Future applications may involve backfilling crushed salt around heat generating waste packages where near-field temperatures could reach 250°C or hotter. The ability to model the reconsolidation of the crushed salt backfill under elevated temperatures is crucial to the determination of both the short-term and the long-term performance of the underground repository.

The current constitutive model for crushed salt reconsolidation accounts for the effects of moisture, through pressure solution, and dislocation creep. It is based on the multi-mechanism deformation (M-D) model used for intact salt [1]. The crushed salt model is dependent on an effective stress to account for the effects of pore porosity. In the limit of zero moisture content, the pressure solution term contributes nothing to the strain rate and at the limit of zero porosity the dislocation creep term becomes equal to the M-D model for intact salt [2]. The M-D model utilizes an exponential temperature dependence in the formation of an activation energy for each deformation mechanism [1].

There has been little experimental work done on crushed salt at higher temperatures necessary to thoroughly dry the salt, so currently it is not possible to validate the predicted elimination of the pressure solution term. Temperatures significantly above 100°C may drive off all surface-bound water, which should eliminate pressure solution effects. There may be some water trapped in the fluid inclusions found in the salt, but it is expected to remain trapped and thus ineffective in modifying the reconsolidation behavior. Furthermore, the activation energies of the deformation mechanisms have not been validated at temperatures above 100°C.

A series of laboratory experiments was conducted to investigate the effects of hydrostatic stress on dry, run-of-mine salt at temperatures up to 250°C [3]. The experiments were chosen to provide crushed salt reconsolidation data that was not previously available. The results of the experiments were compared to analogous calculations using the crushed salt model [4]. Comparisons of the experimental data with the modeling results were used to evaluate the capabilities of the current crushed salt model.

## 2. EXPERIMENTS

Samples were constructed using run-of-mine salt obtained from the Waste Isolation Pilot Plant. Sufficient

material was obtained to allow all planned tests to be carried out using the same batch of material. All the salt was passed through a 9.5 mm sieve and then dried for several days at 105°C until the weight loss stabilized. The crushed salt was placed in right circular cylinder jackets with a 10 cm outer diameter and 20 cm length (see Figure 1) [3].



Figure 1. Crushed salt in lead jacket before compaction [3].

Initially, the jacket consisted of an inner lead liner and an outer Viton layer, but the Viton material was found to be unacceptable at temperature in excess of 100°C. The Viton material used in the experiments appeared to lose nearly all its tensile strength at about 100°C. When used for testing intact specimens, such as cored rock salt, the effect is not important because the deformations are smaller. The large strains in the crushed salt reconsolidation experiments caused the Viton material to fail, which resulted in the confining fluid penetrating into the specimen. Using a second outer lead jacket instead of the Viton material produced successful results. A single thicker lead jacket could have been used as well. It appears that these results represent the first successful quasistatic tests on crushed salt at temperatures to 250°C and pressures up to 20 MPa [3].

All tests were conducted under hydrostatic pressure using a triaxial testing system [5]. Samples were suspended from the upper vessel closure and vented through a nipple in the upper end cap. All heated tests began by loading the jacketed specimen into the vessel, filling the vessel with silicone oil and flushing oil through the vessel to remove trapped air. The oil-filled-vessel was then pre-heated to the planned test temperature. Any moisture reabsorbed onto the previously-dried salt was assumed to be driven off by the pre-heat step which typically lasted 12-18 hours to ensure the sample and oil reached thermal equilibrium [3].

Quasistatic hydrostatic compression tests, designed to look at the short-term response of the salt to increasing pressure, were done by advancing the ram into the vessel while the confining fluid pressure, ram displacement and fluid temperature were recorded. As the ram entered the sealed pressure vessel, the confined fluid was pressurized. Because the sample was not in contact with the ram, it was subjected to pure hydrostatic stress. The experiments were terminated after the desired pressure was obtained.

Density or, equivalently porosity, of the consolidating salt was the key physical parameter to be determined. However the nature of the starting material, inhomogeneous with large particle sizes, makes density a difficult parameter to measure accurately and continuously during a test. Instead, the samples' volumetric change was measured. For all tests the sample's volume change was determined using both the displacement of the main ram and by measurement of expelled air.

Volume changes in the sample reduce the porosity and result in air being expelled from the sample through the vent to the atmosphere. A volume calculation using a spirometer was conducted and used as a check to the volume calculation derived from the main ram displacement. A spirometer is designed to measure gas volume directly for low pressure sources, i.e. lungs. Thus all that is required to use the spirometer to measure changes in porosity is to connect the spirometer to the out-gas vent of the sample. At temperature of 175°C or lower, the volume change measurements using the spirometer and ram methods agreed well, but at 250°C there appeared to be a systematic error in the spirometer [3].

Correct values for the evolving bulk modulus are required to properly model the crushed salt interaction with its surroundings. Because the salt density increased from about 65 to 90% of its theoretical value and the grain-to-grain contact areas changed significantly, the modulus was expected to increase by a large factor. An accepted approach to determining the moduli of

geomaterial matrices is to assume that deformations that occur during unloading (stress paths within the yield surface) are elastic and thus the generic slope of pressure versus volumetric strain is an elastic modulus. Under hydrostatic conditions, the bulk modulus can be determined by measuring the volume strain during a depressurize-repressurize cycle. Several pressure cycles were done at appropriate points during the course of each experiment. Figure 2 shows the pressure versus volumetric strain for the test at 100°C.

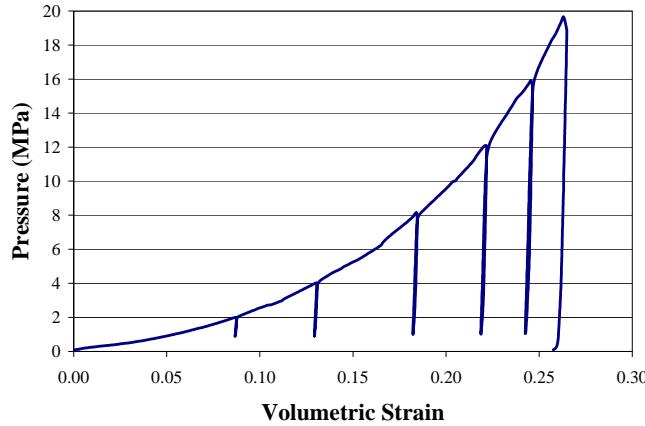


Figure 2. Pressure versus volumetric strain for a crushed salt sample at 100°C.

Eight quasistatic tests produced useful results although only three of them could be considered a full success, meaning that the desired pressure and temperature was reached without a jacket or other failure. The initial and final porosity values, temperature and maximum pressure for each of the quasistatic tests is shown in Table 1.

Table 1. Summary of experimental results.

Temperature (°C)	Maximum Pressure (MPa)	Initial Porosity	Final Porosity
100	20.0	0.35	0.11
175	4.6*	0.36	0.22
175	6.5*	0.35	0.17
175	6.8*	0.37	0.19
175	19.7	0.37	0.10
250	1.2*	0.38	0.31
250	10.6*	0.39	0.12
250	14.9	0.38	0.09

\*Failure before test completion

Systematic increases in compressive volume strain as test temperature increased are readily observed (Table 1). Figure 3 shows a compacted sample tested at 250°C. This sample reached a final porosity of 12% and was cut into quarters for further analysis. All tested specimens were preserved for post-test microscopic examination

and analysis to characterize salt reconsolidation processes in future work [3].



Figure 3. Post experiment crushed salt specimen compacted at 250°C.

Figure 4 shows the measured bulk modulus versus volumetric strain for the three test temperatures. The bulk modulus increased steadily with volumetric strain from a starting value of about 1 GPa up to about 7 GPa. It appears that independent of temperature, it is compaction that determines the bulk modulus, which increases linearly with volumetric strain.

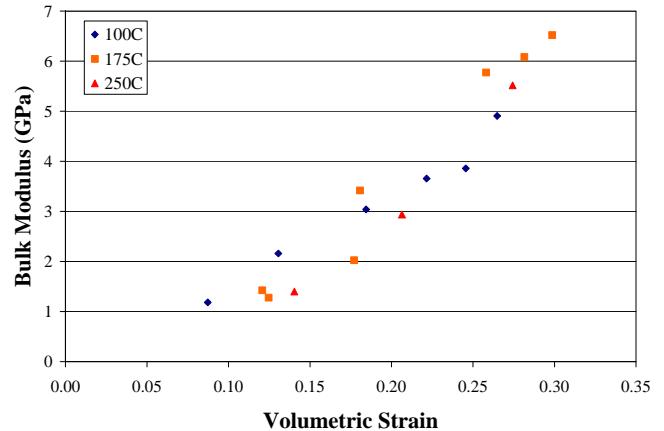


Figure 4. Bulk modulus versus volumetric strain for crushed salt samples at 100°C, 175°C and 250°C.

### 3. MODELING RESULTS

The results of the experiments were compared with analogous modeling results. By comparing the modeling results at elevated temperatures to the experimental results, the adequacy of the current crushed salt reconsolidation model was evaluated. The pressure and temperature measurements as a function of time from each experiment were used in the modeling. This was especially important for the pressure, as some tests included several depressurize-repressurize cycles during which compaction rates were significantly lowered.

Figure 5 shows the measurements of porosity of a quasistatic test ending at 20 MPa and maintained at 100°C compared with the modeling results simulating the experiment. The green dashed line shows the pressure history, including the depressurize-repressurize cycles which were performed throughout the test. The experiment shows the porosity decreasing from 35% to 11%, while the model shows the porosity decreasing to 12%. The modeling results track with the trends shown in the 100°C experimental results [4].

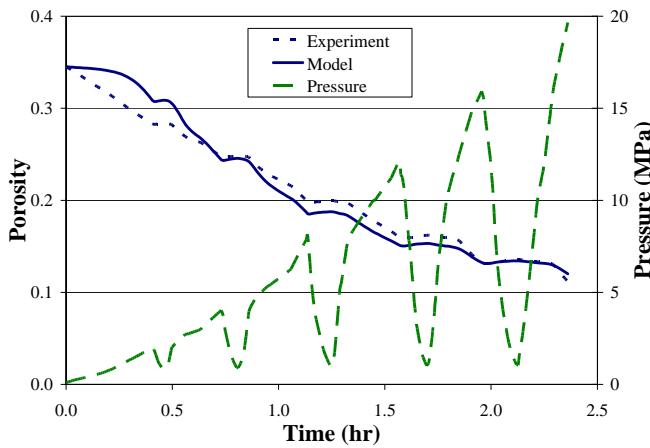


Figure 5. Experimental measurements of porosity versus time for a crushed salt sample at 100°C compared with modeling results of the experiment.

Figure 6 shows the measurements of porosity of a quasistatic test ending at 19.7 MPa and maintained at 175°C compared with the modeling results simulating the experiment. The pressure history shown as the green dashed line indicates that depressurize-repressurize cycles are only present in the second half of the test. This is because the depressurize-repressurize cycle data were collected at the lower pressures for the three partially successful tests at 175°C (see Table 1). The experiment shows the porosity decreasing from 37% down to 10%, while the modeling shows the porosity decreasing to 9%. The difference between the experiment and model increases in the first half hour and then decreases as the experiment progresses [4]. The

decrease in the difference between the experiment and model is mainly due to the end result of zero porosity.

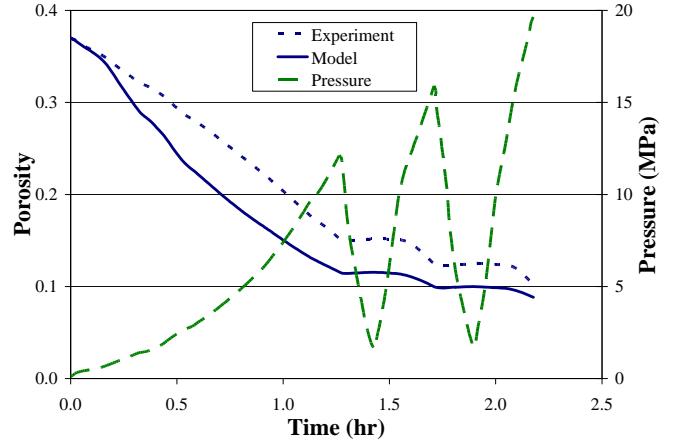


Figure 6. Experimental measurements of porosity versus time for a crushed salt sample at 175°C compared with modeling results of the experiment.

Figure 7 shows the measurements of porosity of a quasistatic test ending at 14.9 MPa and maintained at 250°C compared with the modeling results simulating the experiment. The pressure history shown as the green dashed line indicates that no depressurize-repressurize cycles were performed for this test. This is because the depressurize-repressurize cycle data at lower pressures were collected in the two partially successful tests at 250°C (see Table 1). The experiment shows the porosity decreasing from 38% down to 9% after two hours, while the modeling shows the porosity decreasing to 7%. The difference between the experiment and model increases in the first hour and then decreases as the experiment progresses [4]. The decrease in the difference between the experiment and model is mainly due to the end result of zero porosity.

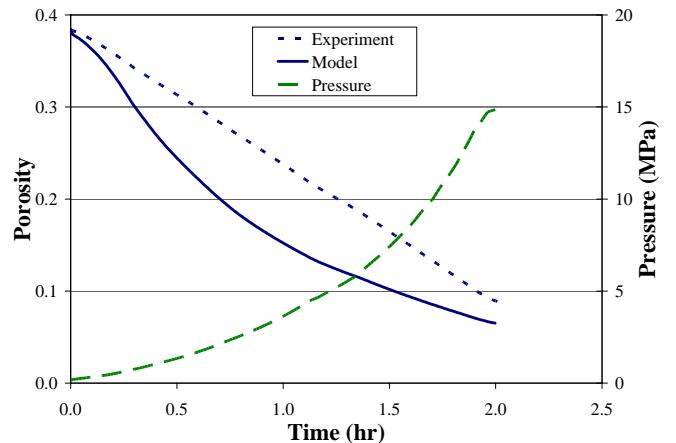


Figure 7. Experimental measurements of porosity versus time for a crushed salt sample at 250°C compared with modeling results of the experiment.

As shown by Figure 5 through Figure 7, the modeling and experimental results both show an increase in the reconsolidation rate with temperature. The modeling results match the crushed salt reconsolidation well at a temperature of 100°C. The crushed salt model over predicts the temperature dependence of the reconsolidation. Further development of the deformation mechanism activation energies would lead to a better prediction of the temperature dependence by the model [4].

#### 4. SUMMARY AND CONCLUSIONS

A series of experiments were conducted to investigate the effects of hydrostatic stress on run of mine salt at temperatures up to 250°C and pressures to 20 MPa. Measurements of bulk modulus show that independent of temperature, it is the degree of compaction that determines the bulk modulus. Tested specimens were preserved for post-test microscopic examination and analysis to characterize salt reconsolidation processes in future work. The results of these tests were compared with analogous modeling results. By comparing the modeling results at elevated temperatures to the experimental results, the adequacy of the current crushed salt reconsolidation model was evaluated. The modeling and experimental results both show an increase in the reconsolidation rate with temperature. The current crushed salt model predicts the experimental results well at a temperature of 100°C and matches the overall trends, but over-predicts the temperature dependence of the reconsolidation. Further development of the deformation mechanism activation energies would lead to a better prediction of the temperature dependence by the crushed salt reconsolidation model.

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