

## **Effect of Curing Temperature and Composition on the Performance Properties of Saltstone – 12473**

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### **ABSTRACT**

When freshly mixed Saltstone is placed into a vault at the Savannah River Site, the heat of hydration produced during curing causes the temperature of the grout to increase. It is not uncommon for the temperature of the grout to increase by 30 to 50 °C above the starting temperature depending on the vault size and location, environmental conditions and pour schedule. For example, temperatures measured by thermocouples in one cell have exceeded 70 °C. The elevated temperatures, once reached, can extend for weeks or even months. Recent studies at Savannah River National Laboratory (SRNL) have shown that curing simulated Saltstone mixes at elevated temperatures can lead to reduced compressive strength and dynamic Young's modulus as well as increased total porosity when compared to the same grout mixes cured at 20 °C. Samples cured at 60 °C can have hydraulic conductivities approximately 600 times greater than samples cured at 20 °C. Previous studies have shown that the 60 °C cured mix had a dynamic Young's modulus that was half that of the mix cured at 20 °C. However, high temperature curing may not always have negative effects on the performance properties of the grout. These results indicate that higher curing temperatures can significantly alter the performance properties that are inputs for the Saltstone Performance Assessment.

### **INTRODUCTION**

The Saltstone Production Facility (SPF) receives low level waste (LLW) salt solution from Tank 50H for treatment and disposal. Tank 50H receives transfers from the Effluent Treatment Project (ETP), the H-Canyon General Purpose Evaporator, and the Actinide Removal Process/Modular Caustic Side Solvent Extraction Unit (ARP/MCU) Decontaminated Salt Solution Hold Tank (DSS-HT). At the SPF, the LLW is mixed with premix (a cementitious mixture of portland cement (PC), blast furnace slag (BFS) and Class F fly ash (FA)) in a Readco mixer to produce fresh (uncured) Saltstone that is transferred to the Saltstone Disposal Facility (SDF) vaults [1]. The Saltstone formulation (mix design) must produce a grout waste form that meets both placement and performance properties. In previous simulated Saltstone studies [1,2], multiple compositional factors were identified that drive the performance properties of Saltstone grout made from the projected ARP/MCU salt solution. This composition was selected as salt solution simulant since ARP/MCU is the primary influent into Tank 50H. The primary performance property investigated was hydraulic conductivity since it is a variable input property to the Saltstone Performance Assessment (PA). In addition, the porosity, also referred to as void structure, is another variable that impacts the PA response. In addition, Young's modulus and cured density are other performance properties analyzed in this report ; however they are indicators of the performance of Saltstone grout and not direct inputs into the PA.

The data showed that the largest impact on the performance properties of Saltstone was due to curing temperature, followed by aluminate concentration in the salt solution, water to premix ratio and premix composition [1,2]. However, due to the scope of the previous studies, only a

few mixes were cured and analyzed at higher temperatures. The samples cured at 60 °C had an increased hydraulic conductivity of approximately 600 times that of the sample cured at room temperature [2]. The hydration reactions initiated during the mixing of the premix and salt solution continue during the curing period in the vaults to produce the hardened waste form product. The heat generated from exothermic hydration reactions leads to a temperature increase in the vaults that depends on the composition of the decontaminated salt solution being dispositioned as well as the grout formulation (mix design). This heat generation is a contributing factor to the temperature increase in the vaults that leads to an increased cure temperature for the grout.

The impact of curing temperature on Saltstone performance properties (hydraulic conductivity, Young's modulus, porosity, etc.) over a range of aluminate concentration, water to premix ratio (w/p) and weight percent fly ash in the premix processed at the SPF will be investigated. The three curing temperatures selected were chosen to provide data at fixed cure temperatures that represent measured temperatures in the SDF vaults. This does not represent the conditions in the vault where the temperature of the Saltstone is continually changing with time. For example, it may take several days for the Saltstone to reach 60 °C at a given elevation. Previous results demonstrated that the rates at which a selected curing temperature is reached affect the performance properties [3]. The approach taken in this task, a rapid increase to the curing temperature, may be conservative with respect to decreased performance. Nevertheless, the data will provide a basis from which to determine the impact of curing temperature on Saltstone performance as a function of key variables [4]. A statistical evaluation of the results for these mixes will be performed to provide the range, and associated uncertainties, of hydraulic conductivity and other properties over this factor space.

The dynamic Young's modulus was originally investigated as a performance property indicator [5] due to the lack of available equipment to measure the hydraulic conductivity of Saltstone samples. Throughout this study, references to Young's modulus are the meant as dynamic Young's modulus. As Saltstone variability studies progressed, the capability to measure hydraulic conductivity became available; however, measuring this property is time intensive and expensive for a large number of samples. In addition, the need for a quick and inexpensive measurement of the performance of the grout still existed. Previous reports [2,3,6] have indicated a relationship exists between these two Saltstone performance properties. The results from previous studies will be combined with the results from this study and statistically assessed. The hydraulic conductivity and dynamic Young's modulus values will be evaluated to determine whether a correlation can be established for these properties and therefore Young's modulus can be used as an indicator of the performance of Saltstone grout samples.

## **METHOD**

A statistically designed set of mixes was developed to allow for the investigation into the key process and compositional factors that affect the performance properties of Saltstone grout. There are eight baseline mixes that contain high and low values of aluminate concentration, varying w/p ratio, and varying fly ash content in the premix. Each of the eight mixes was cured at 22, 40, and 60 °C. Three additional reference mixes were batched at the beginning, middle and end of testing to measure reproducibility. A total of 27 mixes were batched and tested (Table I). Mix numbers 1, 14, and 27 are the reference mixes and are at the mid-points of the compositional and process properties tested. The batch order (run number) for the mixes was chosen at random. The reference mixes were run at the beginning, middle, and end of experiment and used as an indicator of repeatability of batching and analysis methods.

Table I. Experimental Design for Saltstone Mixes Tested

Mix Number	Aluminate Molarity	w/p	Fly Ash (wt %)	Curing Temp (°C)
1	0.165	0.60	47.5	40
2	0.05	0.55	45	20
3	0.05	0.55	45	40
4	0.05	0.55	45	60
5	0.05	0.65	45	20
6	0.05	0.65	45	40
7	0.05	0.65	45	60
8	0.05	0.55	50	20
9	0.05	0.55	50	40
10	0.05	0.55	50	60
11	0.05	0.65	50	20
12	0.05	0.65	50	40
13	0.05	0.65	50	60
14	0.165	0.60	47.5	40
15	0.28	0.55	45	20
16	0.28	0.55	45	40
17	0.28	0.55	45	60
18	0.28	0.65	45	20
19	0.28	0.65	45	40
20	0.28	0.65	45	60
21	0.28	0.55	50	20
22	0.28	0.55	50	40
23	0.28	0.55	50	60
24	0.28	0.65	50	20
25	0.28	0.65	50	40
26	0.28	0.65	50	60
27	0.165	0.60	47.5	40

The nominal premix distribution used in this testing is 45 wt % slag, 45 wt % thermally beneficiated Class F fly ash and 10 wt % portland cement. For those mixes batched using 50 wt % fly ash, the slag content will be reduced to 40 wt %. The premix materials were received in five gallon containers from the vendors during delivery of the bulk materials to the SPF. The premix materials were maintained to limit exposure to humid air and hydration prior to use.

The three curing temperatures selected for this study were chosen to provide data at fixed cure temperatures. This does not represent the conditions in the vault where the temperature of the Saltstone is continually changing with time. For example it may take several days for the Saltstone to reach 60 °C at a given elevation. Previous results demonstrated that the rates at which a selected curing temperature is reached affect the performance properties [3]. The approach taken in this task, with for example, an increase to 60 °C curing temperature may be conservative with respect to decreased performance. Nevertheless, the data will provide a basis from which to determine the impact of curing temperature on Saltstone performance as a function of key variables.

### Sample Preparation

The salt solution and premix materials were mixed for approximately three minutes using a paddle blade mixer. The mixing was paused for approximately five seconds after 30 seconds of mixing to allow entrained air to escape from the grout. Each grout mix (Table I) was batched in duplicate 3200 gram batches to facilitate complete mixing and ease of handling.

Each mix was cured for a total of 90 days before being analyzed. For the first 28 days of the cure cycle, the sample was cured at ambient temperature on a bench top (nominally 20 °C) or in ovens set at either 40 or 60 °C. After 28 days, the samples were removed from the oven and cured at ambient temperature for the remaining 62 days. The samples were weighed after being poured into the cylinders and then again after curing for 90 days.

### **Measurement of Grout Properties**

Fresh properties were measured after the grout had mixed for the designated three minutes. These properties include: yield stress, plastic viscosity, bleed water volume, gel time and set time [7]. Rheological properties were measured using a Haake M5/RV30 rotoviscometer. The flow curves for the mixes were fitted to the Bingham Plastic rheological model to determine the yield stress (Pa) and plastic viscosity (cP). The bleed water volume was measured on duplicate samples of fresh grout placed in sealed cylinders and left untouched for 24 hours.

For gel time, fresh grout was poured into a series of cylinders and left undisturbed. Every five minutes, the grout was poured from a cylinder into an empty container. This was repeated until the fresh grout had developed sufficient structure such that it did not flow as a result of its own mass. The time at which the grout did not flow from the cylinder was designated as the gel time. A Vicat needle was used to determine the final set times [7] twice daily.

The dynamic Young's Modulus (E) was measured according to ASTM standard C 215 [8] using an E-Meter Mk II Resonant Frequency Tester by James Instruments, Inc. The method involves a longitudinal impact on the end of a 3 x 6 inch cylinder of cast and cured paste, detection of the sound waves produced at the opposite end of the cylinder, and measurement of the fundamental resonance frequency of the cylinder through a fast Fourier transform of the time domain signal. Using this resonance frequency and the independently measured mass and dimensions of the cylinder, the dynamic Young's modulus was calculated [8].

The porosity was determined by the mass loss upon heating samples (1.5 to 2 grams) to 105 °C using a Mettler Toledo HR83 Moisture Analyzer. This instrument measures mass loss as a function of time until no further mass loss is observed. The typical time for measurement is on the order of 30 minutes but can be up to one hour if the cured grout is not broken into smaller pieces. The grout pieces that were used in the measurement of porosity were taken from the center of the cylinder by breaking the cylinder and removing pieces from the center region.

The Young's modulus [8], moisture retention [9], porosity, and density of the cured samples were tested at SRNL. In addition, the saturated hydraulic conductivity [10], moisture retention, dry bulk density, and porosity were measured by an offsite laboratory. The saturated hydraulic conductivity of the Saltstone samples was determined by ASTM D 5084 method F, the constant-volume falling head using a flexible wall permeameter [10]. The fluid used for testing was the low aluminate salt solution. This was used to avoid negative interactions of the test fluid and the sample during testing. Saturated hydraulic conductivity is a function of the porous medium and the properties of the test fluid as described by Darcy's law [11].

The fresh properties of the grout mixes are important to ensure the product can be processed through the facility and pumped to the SDF vault (Table II). In the vault, the grout should be formulated such that it sets within three days and produces less than three volume percent of bleed water. The gel time of the fresh grout should be between 20 and 60 minutes. A gel time of less than 20 minutes limits the workability of the grout during a process upset while it is in the facility and longer than 60 minutes can lead to settling and segregation of the grout. The specified gel range is to ensure processability through the facility and that sufficient microstructure develops once the grout is placed in the vault. Development of the structure over a short time period helps prevent segregation of the grout components. Bleed water on top of the set grout is an indication that segregation is occurring [11]. The surface of samples 18 and 24 were still wet after one day but there was not enough bleed to measure. In addition to no bleed, the all of the grouts gelled within 60 minutes, which indicates there was no segregation occurring in the samples prior to microstructure development.

Table II. Fresh grout properties for the 27 grout mixes.

<b>Mix Number</b>	<b>Gel (min)</b>	<b>Fresh Density (g/mL)</b>	<b>Plastic Viscosity (cP)</b>	<b>Yield Stress (Pa)</b>
1	35	1.725	113.70	6.07
2	30	1.737	172.90	9.36
3	20	1.745	NM	NM <sup>a</sup>
4	20	1.735	202.20	10.82
5	45	1.682	79.40	4.37
6	35	1.686	94.85	4.98
7	35	1.692	87.94	4.73
8	30	1.733	135.40	6.88
9	45	1.730	133.30	6.39
10	25	1.726	135.80	6.52
11	50	1.680	65.22	3.20
12	60	1.679	65.22	3.08
13	40	1.679	72.09	3.46
14	30	1.698	106.50	4.75
15	30	1.735	167.10	7.55
16	20	1.745	155.10	9.20
17	20	1.751	168.00	8.10
18	35	1.692	70.65	3.10
19	40	1.697	74.68	3.31
20	35	1.693	72.54	3.72
21	25	1.721	135.40	6.88
22	25	1.726	114.90	4.78
23	25	1.726	114.90	4.67
24	50	1.691	56.42	2.03
25	40	1.693	57.66	2.20
26	55	1.685	55.93	2.10
27	30	1.736	75.08	3.67

<sup>a</sup> NM – not measured

## RESULTS AND DISCUSSION

### Young's Modulus

Young's modulus, also known as the modulus of elasticity, is a measure of the stiffness of a material and is defined as the tensile stress divided by the tensile strain [13]. Stiffer (or less elastic) materials have higher values of Young's modulus,  $E$ . Young's modulus has units of pressure which in this report are expressed as giga-pascals (GPa). Cement-based materials such as concrete have  $E$  values in the range of 5 – 30 GPa with the actual values depending upon degree of hydration, the w/p ratio, and other factors.

Young's modulus as a function of cure temperature is shown in Figure I. As shown in previous studies and literature [1,2,14], increasing the cure temperature decreases the Young's modulus of the Saltstone grout. Literature shows that grouts heated to higher temperatures (greater than 60 °C) lose additional moisture than grouts cured at room temperature. This leads to structural decomposition of the hydration products which results in serious loss in durability and strength [11,14,15]. The dehydration of the sample when cured at higher temperatures is evidenced by the mass change between the fresh weight and the cured weight taken at 90 days.

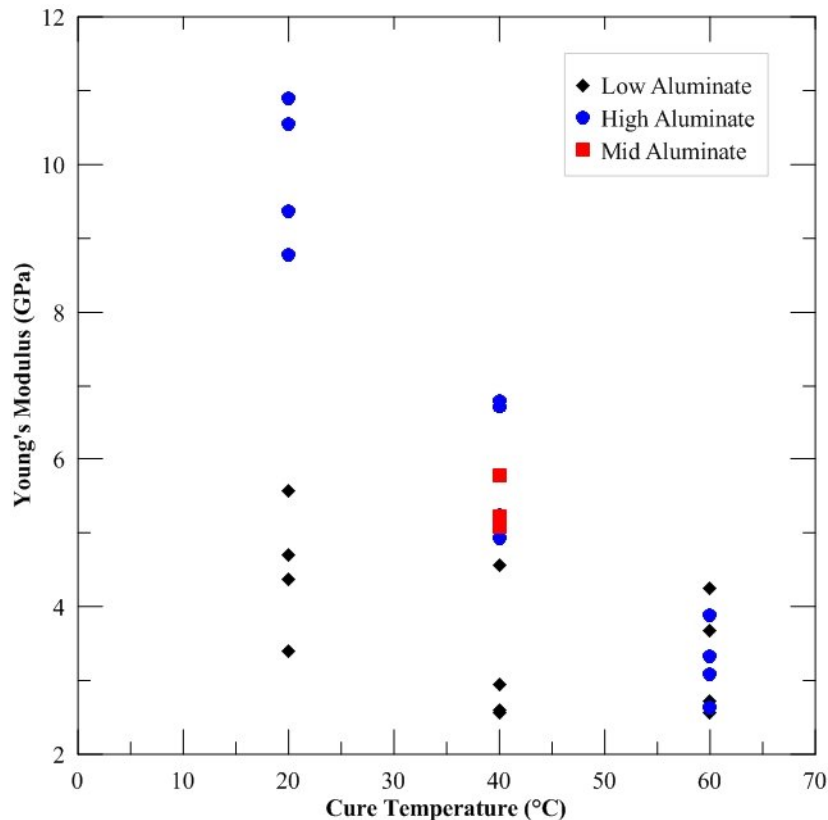


Figure I. Young's Modulus as a Function of Cure Temperature for all Grout Mixes.

The decrease in  $E$  with increasing temperature is also dependent on the aluminate molarity of the salt solution. At 20 °C, the grouts made with the high aluminate solution have higher  $E$  values than the low aluminate grouts. As shown in Figure I, the increased strength due to high

aluminate molarity is severely decreased as the cure temperature increases. The samples formulated with the low aluminate salt solution have E values that are relatively constant across the cure temperature profile as compared to the samples formulated with the higher aluminate solution. At 60 °C, the Young's moduli of all the samples are similar and therefore, the compositional or operational factors of the grout mix do not appear to have an effect.

The strength of concretes and grouts is typically noted by compressive strength measurements and the Young's modulus is used as an indicator of the performance properties since there are multiple factors that can affect the E values of a grout sample. The elastic modulus of a grout matrix is primarily determined by its porosity [11]. One of the primary factors that control the porosity of grouts is the water to premix ratio. In a previous study, it was demonstrated that the Young's modulus increases with decreasing w/p ratio [5]. This trend is confirmed by the results of this current study.

### Hydraulic Conductivity

Permeability is defined as the property that governs the rate of flow of a fluid into a porous solid. For steady-state flow, the coefficient of permeability, also known as hydraulic conductivity, is determined by Darcy's equation [11]. The saturated hydraulic conductivity was determined after the sample had cured 90 days. As discussed with Young's modulus, the increased temperature can lead to decreased durability and strength. One proposed mechanism for the lowered performance properties as a result of increased temperature is microcracking [15] which would result in a higher measured hydraulic conductivity.

The samples made with the low aluminate simulant have generally higher hydraulic conductivities than the samples formulated with the high aluminate solution which is consistent with previous results [2]. At 40 °C, the high aluminate samples have an improved hydraulic conductivity compared to the hydraulic conductivity of the samples cured at 20 °C. However, the opposite occurs for the grouts made with the low aluminate simulant. Further investigation needs to be performed to determine the cause of these results.

It has been shown in literature that the hydraulic conductivity of cement pastes is primarily determined by the size and continuity of pores [11]. One of the primary compositional factors that control the porosity of the Saltstone is the water to premix ratio. Therefore, the hydraulic conductivity should be affected by the water to premix ratio of the grout mix (**Error! Reference source not found.**). There is a general trend that increasing the w/p, increases the hydraulic conductivity of the grout, but the data do not lead to any definite conclusions.

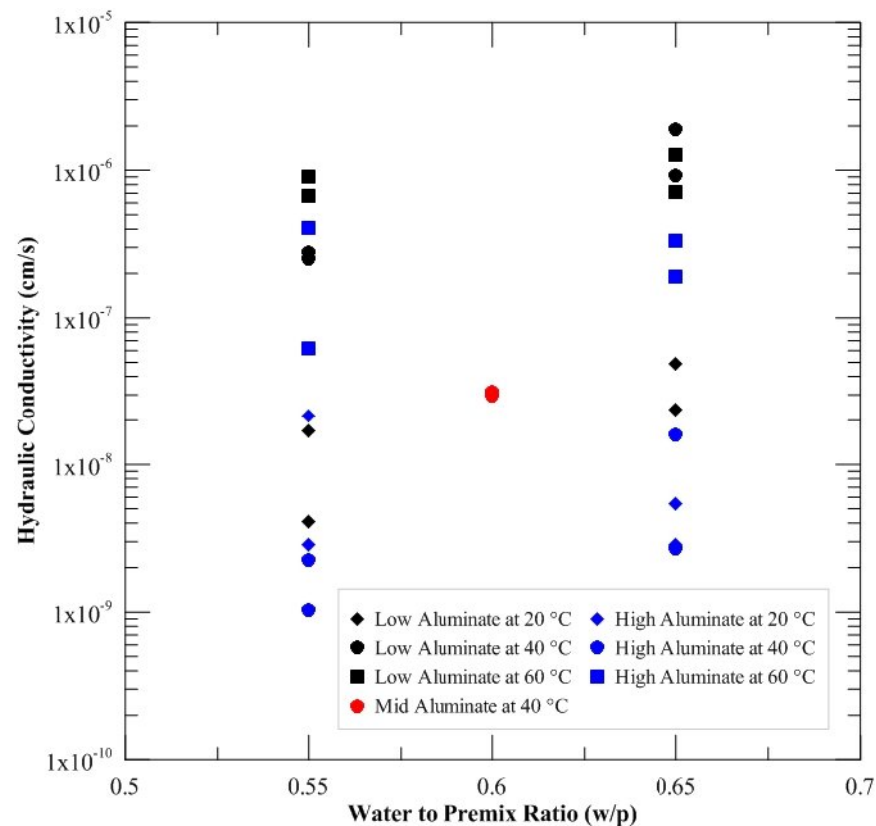


Figure II. Hydraulic Conductivity as a function of w/p for all mixes.

## Porosity

Grout porosity is generally defined as the percentage of total volume of cured grout that is not occupied by either the starting cementitious materials (in this case, portland cement, blast furnace slag, and Class F fly ash) or the products that results from reaction of these cementitious materials with water (calcium silicate hydrate, calcium hydroxide crystals, etc.) [11]. For Saltstone mixes, the pore volume is occupied by a concentrated salt solution.

Figure III shows that the majority of the grouts batched at the lower water to premix ratio correspond with lower porosity values. In general, grouts with higher w/p have increased porosity assuming that the cementitious materials are completely hydrated [11].



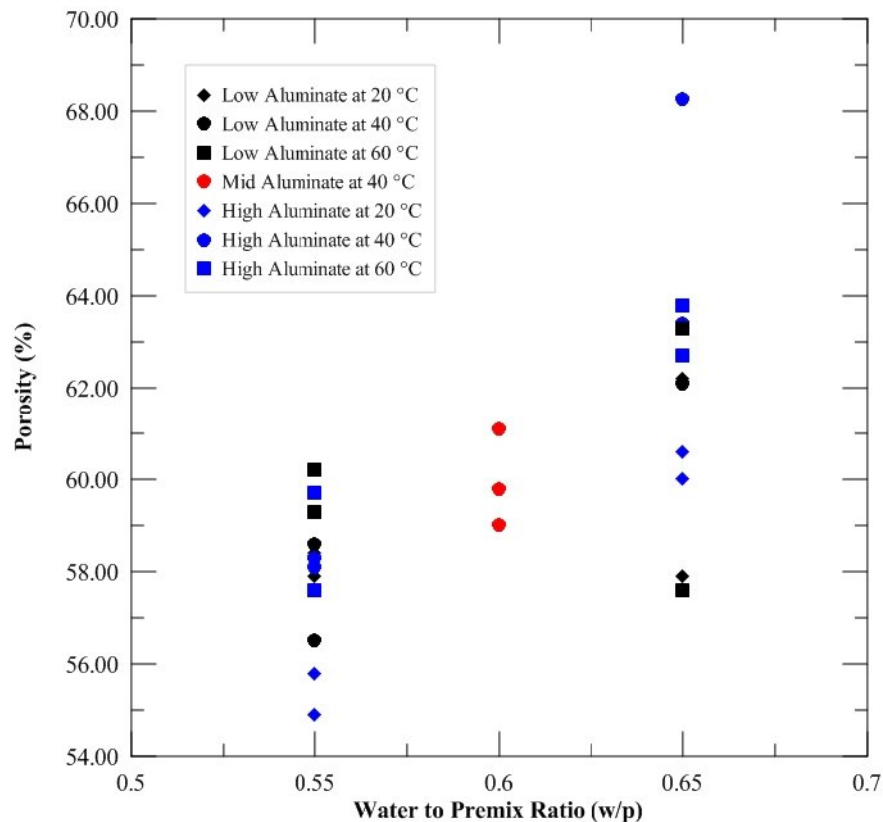


Figure III. Porosity as a function of w/p for all mixes.

### Cured Density

The cured density of the grouts is calculated from the cured weight after 90 days and the length and diameter of a 3x6 inch cylinder. Table II lists the cured densities for each mix in ascending order. The grouts formulated at 0.65 w/p are less dense than the grouts made at 0.55 w/p. Lower w/p mixes have smaller capillary voids than those formulated at higher w/p which results in an overall more dense grout. Grouts formulated at higher water to premix have greater volume expansion due to pore size to accommodate the additional liquid [11]. In addition, the cure temperature has an effect on the density of the grout. The grouts cured at 60 °C have lower densities than the other grouts, which is indicative of drying the sample and loss of pore solution.

Table III. Cured density values listed from lowest to highest and the associated operational and compositional factors for each mix.

Mix Number	Cured Density (g/mL)	w/p	Aluminate Molarity	Fly Ash (wt %)	Curing Temp (°C)
11	1.597	0.65	0.05	50	20
5	1.704	0.65	0.05	45	20
18	1.706	0.65	0.28	45	20
6	1.707	0.65	0.05	45	40
7	1.708	0.65	0.05	45	60
4	1.711	0.55	0.05	45	60
20	1.711	0.65	0.28	45	60
26	1.717	0.65	0.28	50	60
24	1.721	0.65	0.28	50	20
13	1.727	0.65	0.05	50	60
23	1.733	0.55	0.28	50	60
19	1.735	0.65	0.28	45	40
12	1.742	0.65	0.05	50	40
14	1.742	0.60	0.165	47.5	40
21	1.745	0.55	0.28	50	20
27	1.745	0.60	0.165	47.5	40
8	1.747	0.55	0.05	50	20
25	1.747	0.65	0.28	50	40
1	1.755	0.60	0.165	47.5	40
10	1.757	0.55	0.05	50	60
15	1.764	0.55	0.28	45	20
17	1.769	0.55	0.28	45	60
22	1.771	0.55	0.28	50	40
16	1.772	0.55	0.28	45	40
3	1.773	0.55	0.05	45	40
9	1.782	0.55	0.05	50	40
2	1.783	0.55	0.05	45	20

### Correlation Between Dynamic Young's Modulus and Hydraulic Conductivity

Previous studies on the performance properties have indicated a relationship exists between the hydraulic conductivity and Young's modulus for Saltstone [1,2,6]. The limited data set analyzed in those studies indicated a relationship between the two performance properties [6,9,16]. However, incorporating data from a previous study [9] with the data from this study indicates a quadratic relationship (Figure IV). The R-squared value for this fit is 77.1% which indicates that a better fit may be possible with more data points.

There is a linear correlation these performance properties up to Young's modulus values of 7 GPa and hydraulic conductivities of  $1 \times 10^{-9}$  cm/s. At this point, that the data becomes more scattered and the data is not as well modeled. The constant-volume falling head method [10] is known to have difficulty measuring samples with hydraulic conductivities values around  $1 \times 10^{-9}$  to  $1 \times 10^{-10}$  cm/s or greater. This could account for the increased scatter in the data at higher Young's modulus values.

It appears that this model can be used to estimate the hydraulic conductivity of a sample based its Young's modulus for a limited range of values. More data points should be included to further define the relationship between these performance properties are high Young's moduli and low hydraulic conductivities. The Young's modulus should continue to be measured on grouts formulated in future studies and it can be used as screening method for samples to be sent for hydraulic conductivity measurements.

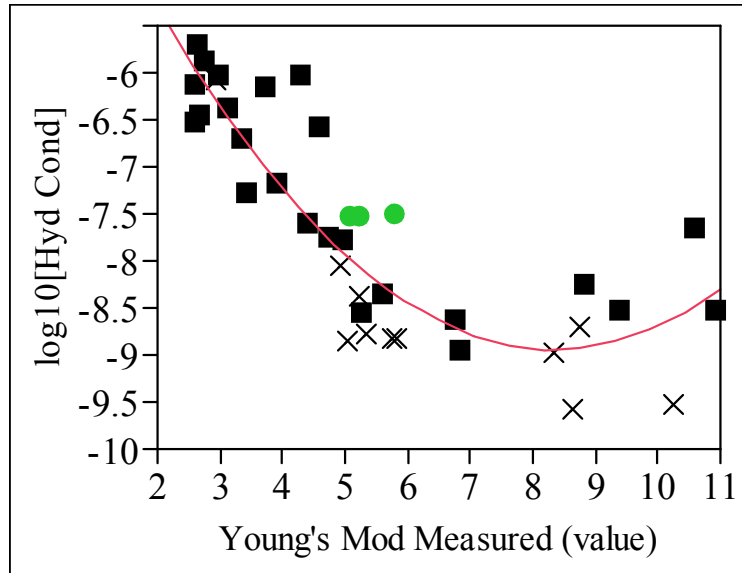


Figure IV. Normal Log of hydraulic conductivity as a function of Young's modulus showing a second degree polynomial line fit.

## CONCLUSIONS AND FUTURE WORK

The Saltstone formulation (mix design) must produce a grout waste form that meets both placement and performance properties. A statistically designed set of mixes was developed to determine key process and compositional factors that affect the performance properties of Saltstone grout. A total of 27 mixes were batched and tested. There are eight baseline mixes that contain high and low values of aluminate concentration, varying w/p ratio, and varying fly ash content in the premix. Each of the eight mixes was cured at 22, 40 and 60 °C. The three additional reference mixes batched at the beginning, middle and end of testing showed good reproducibility throughout the study.

The results of varying the operational and compositional factors on the performance properties of Saltstone grout are:

- The Young's modulus is inversely affected by the curing temperature of the grout.
- The aluminate content of the salt solution increases the Young's modulus of the Saltstone for samples cured at 20 and 40 °C.
- All grouts cured at 60 °C have low Young's moduli and the composition of the grout mix did not have any positive effects on the performance properties after curing at this temperature.
- The fly ash content in the premix had not discernable effect on the Young's modulus.

- The water to premix ratio does not affect the Young's modulus.
- Grouts made with the high aluminate salt solution have lower hydraulic conductivities than those made with the low aluminate simulant.
- Temperature has an inverse effect on the hydraulic conductivity.
- In general, increasing the water to premix ratio and the fly ash content will increase the hydraulic conductivity.
- The porosity and cured density of the Saltstone grout are primarily a factor of the water to premix ratio of the grout formulation.

The correlation between hydraulic conductivity and Young's modulus investigated in previous studies was evaluated further in this report. The current data as well as data from previous studies was fitted by a second degree polynomial function with a 77.1 % R-squared value. This model can be used to estimate the hydraulic conductivity of a sample based its Young's modulus for a limited range (E values up to 7 GPa). More data points should be included to further define the relationship between these performance properties are high Young's moduli and low hydraulic conductivities. The Young's modulus should still be measured on grouts formulated in future studies to be used as screening method for samples to be sent for hydraulic conductivity measurements.

Saltstone grout has a complicated microstructure that is affected by the composition of the salt solution, the dry feed mixture composition, and the conditions under which it cures. The performance properties investigated are interrelated, which can make it difficult to determine individual relationships between various factors. Porosity is a large factor in determining both the Young's modulus and the hydraulic conductivity of the samples. Although the cured density is a straightforward measurement, it is not a significant performance property that needs further investigation. The porosity measurement provides similar and more meaningful data in relation to other performance properties.

Based on the results of this study, the aluminate content of the salt solution does have an effect on the Young's modulus and the hydraulic conductivity and its effects should continue to be investigated. However, a new projection of aluminate contents should be used since projections change over time. The variability of fly ash in the premix did not prove to be a contributing factor that effects the performance properties of Saltstone and doesn't need to be included in future studies unless significant deviations from the baseline mix are proposed.

The water to premix ratio has a significant effect on the performance properties of interest and the effects of varying this factor need further investigation. It is recommended that smaller ranges of w/p are investigated in order to provide data that is more relevant to the Saltstone being placed in the vaults.

Although the cure temperatures used in this study are conservative, it is obvious that curing Saltstone grout at high temperatures has a negative effect on the performance properties. It is recommended for future studies that samples be cured under conditions that more represent the curing conditions found in the vault. This includes cure profiles and curing under high relative humidities. It is unclear if the deleterious effects of curing at high temperatures is solely due to drying of the samples or if there are other effects of curing at elevated temperatures. Curing future sample at various relative humidities will provide further insight into the performance properties of Saltstone.

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