

# Permeability Characteristics of Sand Grouted with Glyoxal Blended Sodium Silicate

**Eyubhan Avci**

Bursa Technical University, Department Civil Engineering, Bursa, TURKEY

## ABSTRACT

The goal of this study was to investigate the permeability characteristics of glyoxal blended sodium silicate injected sand specimens with different relative densities and gradations. Initially, rheological characteristics of glyoxal blended sodium silicate grouts were determined. Accordingly, the gel time decreased as the sodium silicate content increased. Viscosities of glyoxal blended sodium silicate grouts increased as the silicate content increased. Syneresis increased as the silicate content increased up to 37% but then started decreasing as the silicate content continued to increase. The groutability of glyoxal blended sodium silicate grouts into different graded sands specimens formed at various relative densities was highly successful. Grouted sand samples were kept in humidity room at a temperature of 20 °C till test time and put to permeability tests at different time intervals. In general, glyoxal blended sodium silicate grouting decreased the permeability of different graded sand specimens. The permeability of sodium silicate-glyoxal grouted sand specimens increased with time as a conclusion of syneresis.

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**Correspondence to:** Eyubhan Avci, Bursa Technical University, Department of Civil Engineering, Bursa, TURKEY  
Tel: +90 (507) 465-6336  
E-Mail: eyubhanavci@gmail.com

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## INTRODUCTION

Grouting is one of the oldest and widely used ground improvement methods in civil engineering applications all over the world. It is mainly used for reducing permeability and increasing the strength of soil or rock. It is also employed in decreasing the deformability of soil under foundations, stabilizing ground cutting face and excavations and controlling the settlement of ground surface during the opening of a tunnel [1]. Different grouting materials such as suspensions, solutions and emulsions are used for grouting purposes [2-4].

Chemical grouts were developed where the pore size in rock or soil units were too small to be penetrated by conventional Portland cement suspensions [5]. They were fluid at the beginning but reacts after a predetermined time to form a solid, semisolid or gel. Various chemical grouts have been introduced so far, and each one has characteristics suitable for different uses. The most common chemical grouts were introduced by US Army corps of engineers in five categories i.e., sodium silicate, acrylate, lignin, urethane and resin grouts [5].

Sodium silicate grouts, among them, are the most popular ones owing to their safety and environmental suitability. Therefore, the rheology of sodium silicate based grouts and the engineering properties of the grouted soils would be the field of interest. In the past, the hydraulic conductivity of sodium silicate injected sand specimens was studied by Bodocsi and Bowers, Krizek and Spino but their studies were limited to some extent [6,7].

The prime aim of this study was to find out the effect of sand gradation, relative density, and syneresis on the permeability of sodium silicate-glyoxal grouted sand thus contributing to the relevant literature.

## MATERIALS AND METHODS

In this study, Quartz sand taken from Kızılırmak River next to Çorum was used. The specific gravity of sand's was specified to be 2.61 according to ASTM D854-02 [8]. Sand specimen was primarily separated into 2 various subgroups. Both of the subgroups were formed utilizing 2 sets of sieves in a manner that

**Table 1.** Properties of sand specimens used in experimental studies

Sample No	Particle size content (%)		$\gamma_{dry(max)}$ kN/m <sup>3</sup>	$\gamma_{dry(min)}$ kN/m <sup>3</sup>	$e_{max}$	$e_{min}$
	Fine	Medium				
1	100	0	15.80	12.60	1.14	0.71
2	80	20	15.70	12.70	1.13	0.72
3	60	40	15.60	12.70	1.12	0.73
4	30	70	15.50	12.90	1.10	0.74
5	0	100	15.40	13.00	1.08	0.75

Note:  $\gamma_{dry(max)}$ : max. dry density ;  $\gamma_{dry(min)}$ : min. dry density ;  $e_{max}$ : max. void ratio;  $e_{min}$ : min. void ratio

the coarser fraction of sand particles was initially sifted through set of (No 10-No 40) upper sieves. Those remained on a sieve No 40 were collected and defined as medium sand. Thereafter, the other subgroup was sifted through set of (No 40-No 200) lower sieves. The sands particle stayed on a sieve No 200 was collected and defined as fine sand [9]. To widen the range of sand gradation, Fine and medium sands were blended with each other at various percentages by dry mass (Table 1). Thus, five various gradations were obtained and their grain size distributions were shown in Figure 1.

To create different gradations at various relative densities (30%, 50% and 70%), the minimum and the maximum dry unit weights of the specimens were determined in reference with ASTM D4254-00 and ASTM D4253-00 standards respectively (Table 1) [10,11].

### Sodium Silicate, Reactant and Water

Sodium silicate ( $SiO_2 \cdot Na_2O$ ) commercially known as water glass, is available either in an aqueous solution or powder. The silica/alkali ratio used for grouting is generally in the range of 3 to 4. Sodium silicate used in this study is produced by Ege Holding in Turkey with a brand name of EGENat 3203. Its physico-chemical properties were given in Table 2.

Sodium silicate solutions are alkaline so they have to be neutralized to form a gel. For this purpose, the reactant used

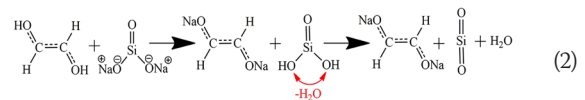
**Table 2.** Physico-chemical properties of Sodium Silicate and Glyoxal

	Coloring	Colorless, clear liquid
Sodium Silicate	Formulation	$Na_2SiO_3$
	Weight modules ( $SiO_2/NaO$ )	3.0-3.3
	Molecular modules ( $SiO_2/NaO$ )	3.1-3.4
	$Be'$ (20 °C)	39-41
	Density (20 °C, gr/cm <sup>3</sup> )	1.37-1.39
	$Na_2O$ (%)	8.5-9.5
	$SiO_2$ (%)	26.0-28.0
	pH (20 °C)	11.66
	Viscosity (20 °C, cP)	75-150
	Coloring	Clear colorless to yellow liquid
Glyoxal	Formulation	$C_2H_2O_2$
	Formula Weight (gr/mol)	58.04
	Density (20 °C, gr/cm <sup>3</sup> )	1.27
	Acetic Acid	0.25% max.
	pH (20 °C)	2.1-2.7

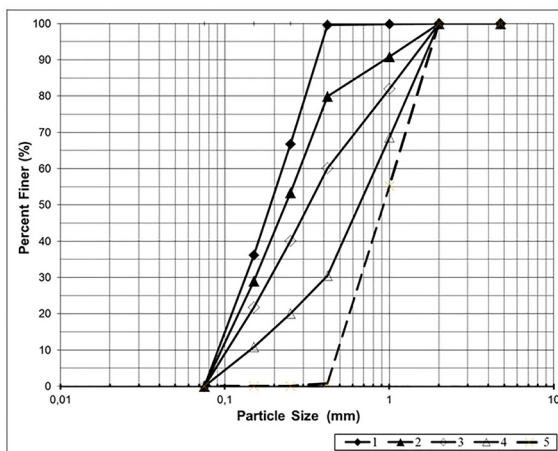
was Glyoxal produced by Acros Organics. The physico-chemical properties of Glyoxal were also given in Table 2. The tap water was the third component in forming grouts.

### Chemical Reaction

Silica is a weak acid, and sodium silicate is thereby basic. Sodium silicate will be precipitated as a gel by neutralization. Firstly, glyoxal ( $C_2H_2O_2$ ) was converted to oxalaldehyde form in acidic medium. Then, a dilute sodium silicate ( $Na_2SiO_3$ ) mixed with organic origin oxalaldehyde ( $C_2H_4O_2$ ) will form a gel with time based on chemical concentrations. The reaction scheme is presented below (Eqs. 1 and 2):



The glyoxal was converted to oxalaldehyde, acidic derivative, by acidic media (Eq. 1). Then the acidic oxalaldehyde were neutralized with basic natrium silicate as shown in Eq. 2. The natrium formate ( $C_2H_2O_2Na_2$ ) salt and silicic acid ( $H_2SiO_3$ ) were then obtained but the silicic acid was unstable in air atmosphere so it converted to silicium dioxide (silica or silicon dioxide) ( $SiO_2$ ) by vomiting one molecule aqua and the mixture gelled.



**Figure 1.** Grain size distribution of sand specimens.

### Sample Preparation and Grouting

The grouting test apparatus consisted of a manometer, 100 molds for hydraulic conductivity, a grout tank with propeller and related connections. Molds were 52 mm in diameter and 120 mm in length. The test apparatus was detailed in Figure 2. The grouting test apparatus was developed by Mollamahmutoglu and Avcı [12].

The internal surfaces of the molds were slightly greased to eliminate specimens' disturbance while the samples were removed from molds after grouting. During specimen preparation, a coarse layer of sand regarding eight mm in thickness was former located at the base of the molds to evenly distribute the grout into the specimens. Specimens were then placed into molds in 3 equal layers. Each layer was compacted utilizing a vibratory hammer to accomplish the required relative density prior to the next one. For 30, 50 and 70 percent relative densities of samples, the first void ratios ( $e_v$ ) were calculated from the empirical relation of relative density since the max. and min. void ratios were obtained by experiments as explained earlier. Afterwards, depending on specific gravity and void ratio of samples, the relevant dry unit weight was figured out and the essential mass of samples was calculated. From every one of these amounts, each layers' relative density was controlled.

Afterwards forming the samples at the required relative density, a coarse layer of sand about eight mm in thickness was put on the upper part of the molds. Next the base and top end-plates of the molds were assembled using tie rods (Figure 2). Lastly, specimens were filled with water. The

**Table 3.** Gyoxal blended Sodium Silicate solution grouts

Solution No	Water (%)	Sodium Silicate (%)	Reactant (%)
SG <sub>1</sub>	67	29	4
SG <sub>2</sub>	56	37	4
SG <sub>3</sub>	46	46	8
SG <sub>4</sub>	36	53	11

bottom and top ends were sealed against water leakage and put aside till the injection date. Before grouting, the samples were saturated by the upward flow of tap water through the bottom under the pressure of 20 kPa. Water was permitted to flow through the sand specimens ensuring that no air bubbles came out from the outlet at the upper of the mold.

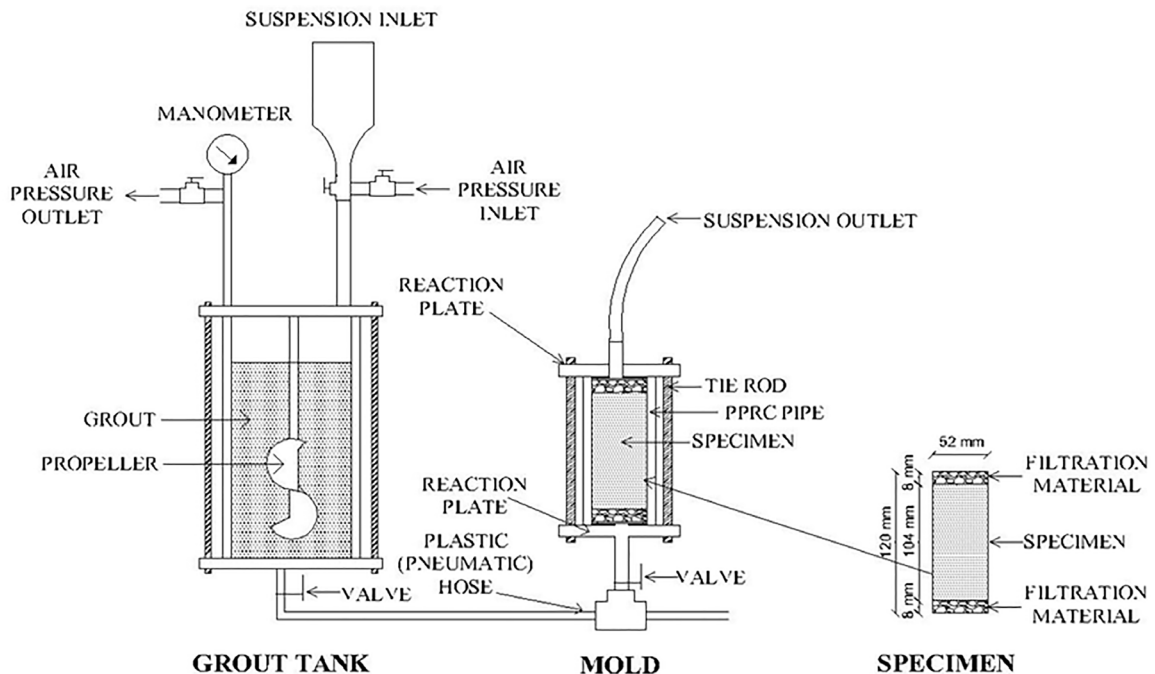
The specimens' grouting pressures were performed by trial and error and the penetration pressures were determined in this direction. The injection pressures was provided with air compressor and observed by a manometer.

Before grouting, water and the pre-measured contents of reactant were blended completely in a holder by an attractive stirrer and after that sodium silicate was then included and the entire solution was mixed again. The grout was poured into grouting tank and was lastly grouted into the samples in molds. The contents of chemical substances forming sodium silicate grouts for this experimental study were presented in Table 3.

### TESTING PROGRAM

#### Gel Time, Viscosity and Syneresis Tests

Firstly, the gel times of sodium silicate solution grouts in Table 3 were determined. They were defined as the



**Figure 2.** The details of grouting test apparatus [12].

elapsed time from grout mixing until no grout flowed out from a baker when tilted to 45° [13]. Grout gels were dependent on the components of the grout. On that account, the effect of silicate content on the gel time of grouts was studied and the findings were presented in Figure 3.

The changes of viscosities of grouts with time were quantified by Brookfield DV-III Ultra Rheometer in reference to ASTM D2196-15 [14] and the results were shown in Figure 4.

For the measurement and observation of the amount of syneresis of grout gels, the grout solutions were preserved in graduated plastic air-tight container. The syneresis and time relation was given in Figure 5. Silicate content effects on the syneresis of grouts were also presented in Figure 6.

### Grout Penetration Test

The penetrability of glyoxal blended sodium silicate solutions into various graded sand specimens at 30%, 50% and 70% relative densities was investigated and the results were shown in Figure 7. The lowest grouting pressures for

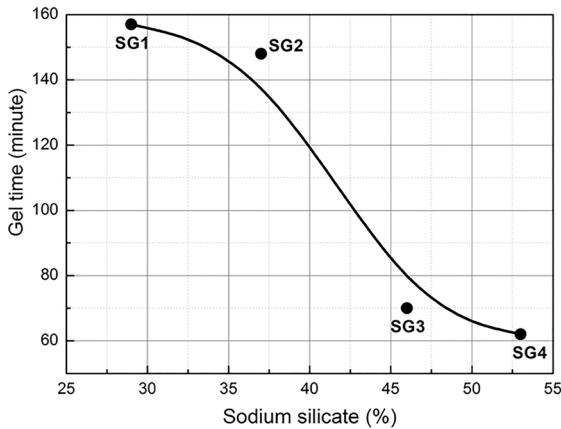


Figure 3. Gel time and Sodium silicate content relation (20°C).

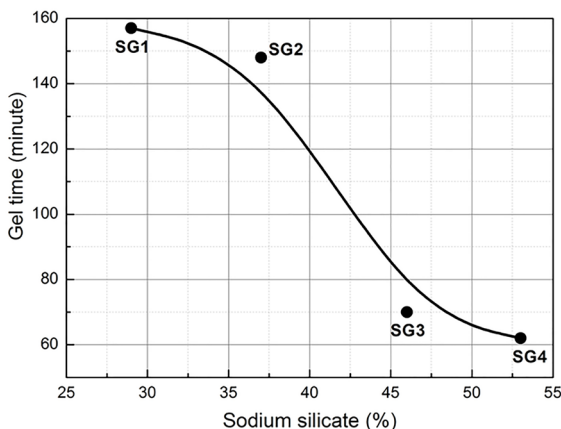


Figure 4. Sodium silicate content effect on the viscosity of grouts (20 °C).

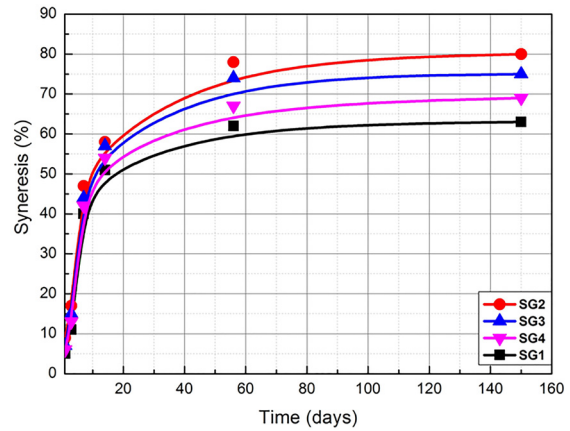


Figure 5. Syneresis of grout gels with time (20 °C).

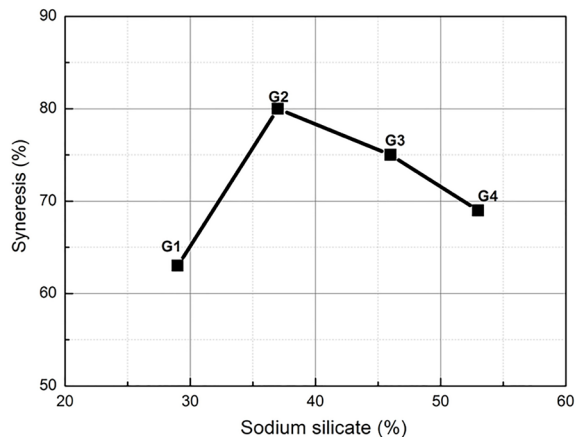


Figure 6. Sodium silicate content effect on the syneresis of grout gels for 720<sup>th</sup> day (20 °C).

the grouting of the sand specimens were also determined by trial and error and presented in Figure 7.

### Permeability Tests

In the beginning, the constant head permeability tests were run on various graded ungrouted sand specimens at various relative densities in accordance with ASTM D2434-68 [15] and their permeability values were measured and given in Figure 8. In addition, the permeabilities of grouted sand specimens with different gradation and relative densities were investigated by conducting falling head permeability test under the gradient of 20 in reference to ASTM D5856-95 [16]. The influences of the curing period and fine sand content on the grouted sand specimen's permeabilities were researched and the related results were presented in Figures 9 and 10.

## RESULTS AND DISCUSSION

### Gel Time Viscosity and Syneresis

The gel time of the grouts was based on the solution concentration. Figure 3 showed that increase in sodium silicate content decreased the gel time of grouts. The gel

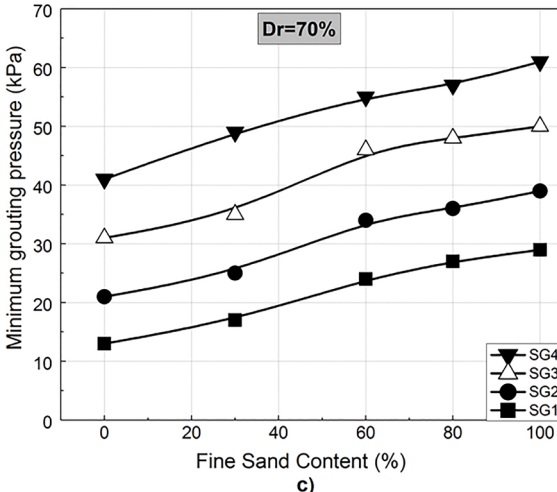
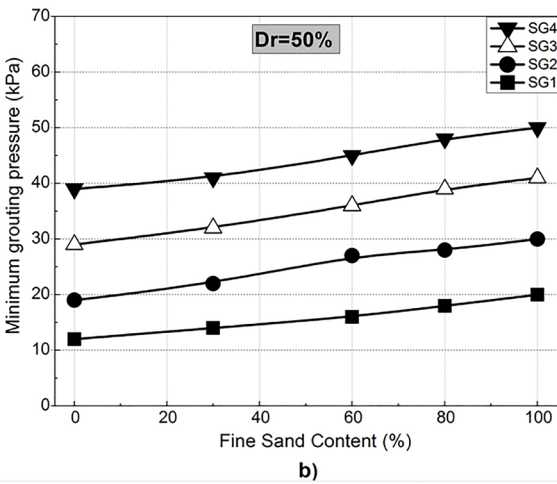
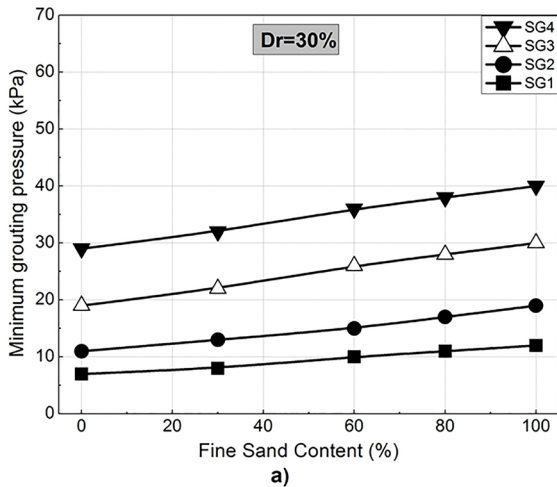


Figure 7. Grouting pressure variation with fine sand content.

time, at a room temperature of 20 °C, decreased from 157 to 62 minutes by changing the silicate content from 29 to 53 percent in return.

The outlet viscosity was increased with the increase in silicate content. The outlet viscosity for the grout of 29% silicate content and 4% reagent mixture was 1.95 cP and it

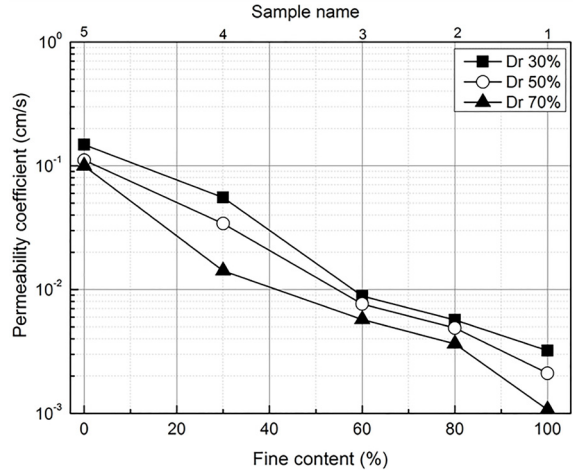


Figure 8. Permeability values of ungrouted sand samples.

was 4.89 cP for the grout of 53% silicate content and 11% mixture (Figure 4).

Syneresis is described as the leakage of liquid from the grout gel and expressed as a percentage of the initial grout volume [17]. The liquid leakage from the grout gels was sighted a period of 150 days. It considerably increased up to 56<sup>th</sup> day and continued at a decreasing rate after 56<sup>th</sup> day (Figure 5). Figure 6 showed that syneresis increased as the sodium silicate content increased up to 37% but then started decreasing as the sodium silicate content increased. The syneresis percentages of grout gels for 29%, 37%, 46% and 53% silicate contents were 63, 80, 75 and 69 respectively.

### Penetrability

Sand samples compacted at 30, 50 and 70 percent relative densities were successfully grouted with all contents of sodium silicate solution. The injection pressures varied from 7 to 61 kPa (Figure 7). Increase in relative density, fine sand content and sodium silicate content increased grouting pressures.

### Permeability

Sand specimens' permeabilities were decreased by two to five orders of magnitude after grouting. As the coefficient of permeabilities of ungrouted sand specimens ranged from  $1.07 \times 10^{-3}$  to  $1.48 \times 10^{-1}$  cm/s, the grouted ones varied from  $3.85 \times 10^{-6}$  to  $1.29 \times 10^{-5}$  cm/s on the 150<sup>th</sup> day after grouting (Figure 9 and 10). Bodocsi and Bowers, Krizek and Spino reported similar findings irrespective of the effects of relative density and syneresis [6,7].

Permeabilities of grouted specimens reduced with the increase of fine content of sand. The average permeability values of SG1 grouted 100% medium and 100% fine sand specimens at 30 percent relative density were  $4.63 \times 10^{-6}$  cm/s and  $9.76 \times 10^{-6}$  cm/s respectively at the end of 150<sup>th</sup> curing period (Figure 10).



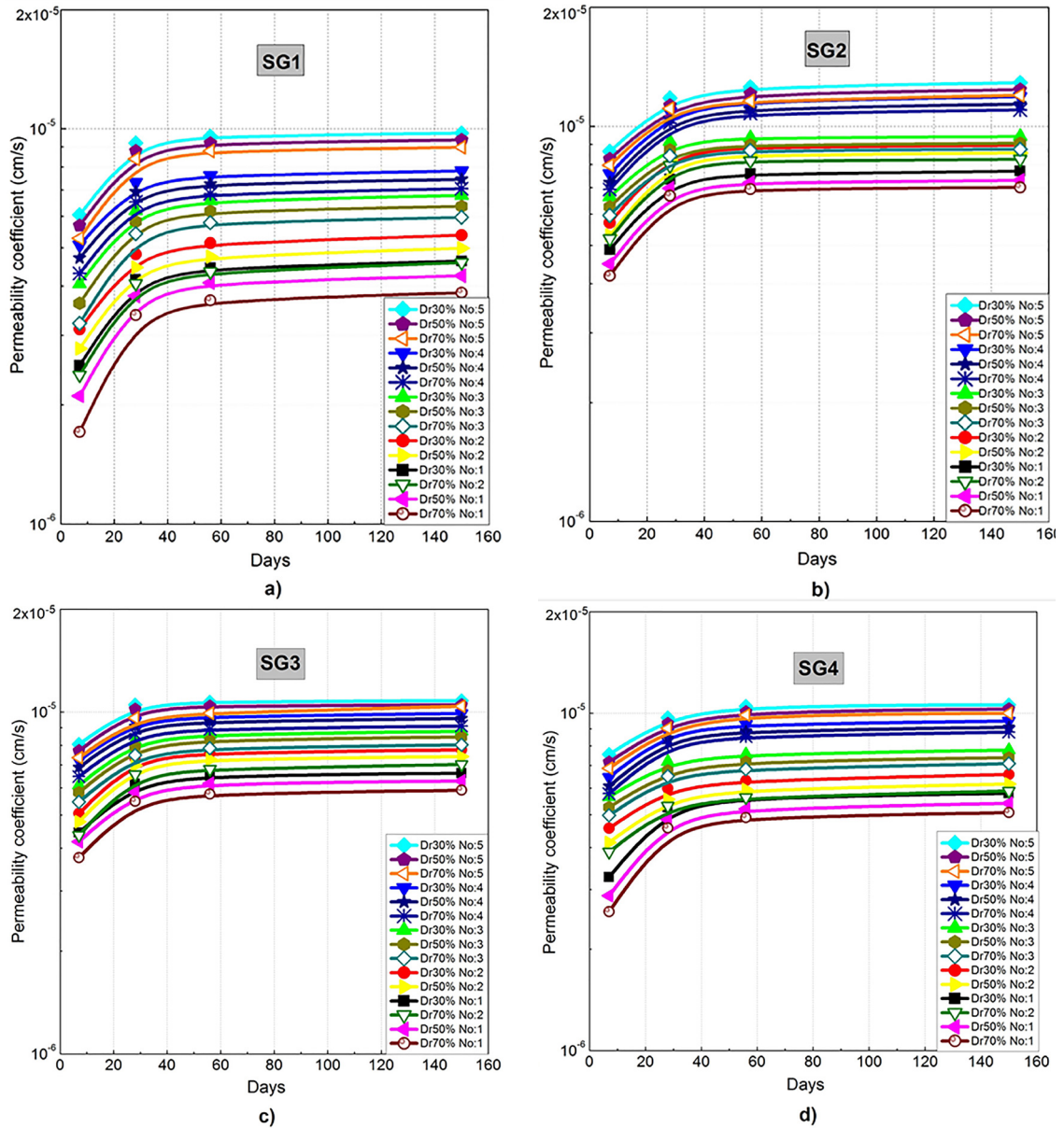


Figure 9. Permeability values of sodium silicate-glyoxal grouted sand specimens for different curing period.

Increase in relative density slightly decreased the permeabilities of grouted sand samples. The average permeabilities of SG1 grouted 100% medium sand specimens at 30%, 50% and 70% relative densities were  $9.76 \times 10^{-6}$  cm/s,  $9.38 \times 10^{-6}$  cm/s and  $8.98 \times 10^{-6}$  cm/s at the end of 150<sup>th</sup> day curing period respectively (Figure 10)

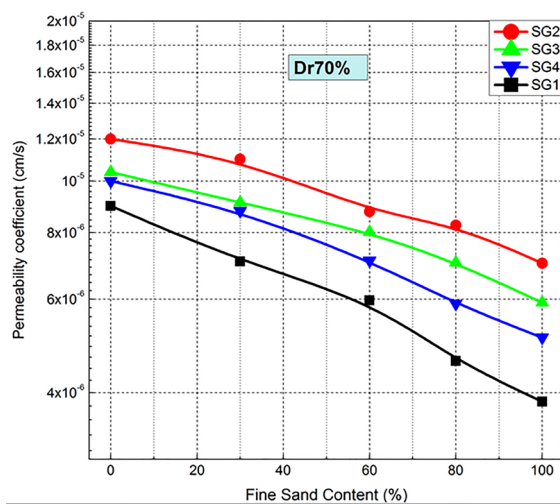
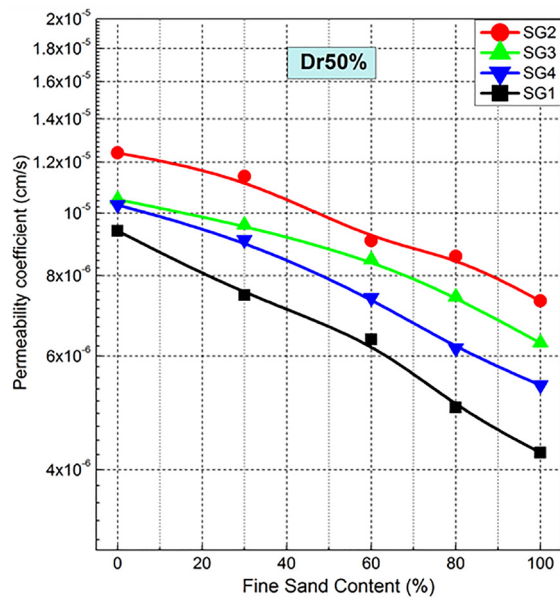
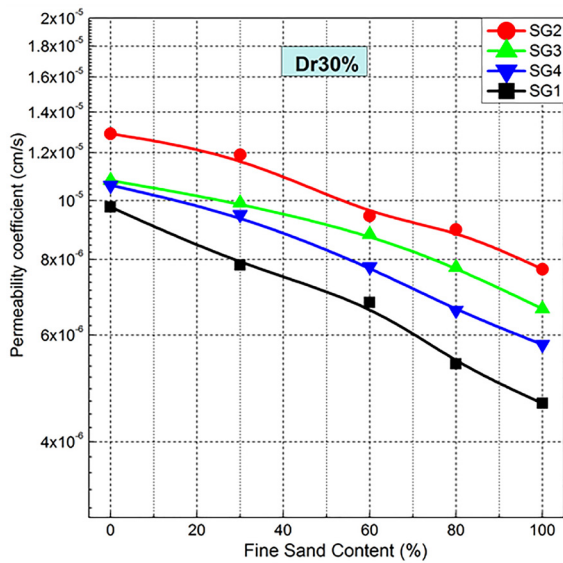
Up to 37 % of silicate content, permeability of injected sand specimens increased with the increase of silicate content and then decreased as the sodium silicate content increased. The permeability coefficients of grouted fine sand specimens at 30 percent relative density were  $4.63 \times 10^{-6}$  cm/s,  $7.71 \times 10^{-6}$  cm/s,  $6.63 \times 10^{-6}$  cm/s and  $5.79 \times 10^{-6}$  cm/s for 29%, 37%, 46% and 53% silicate contents by the end of 150<sup>th</sup> day respectively.

Increase in the permeabilities of injected sand samples was observed with time at a decreasing rate. The average permeability coefficients of SG1 grouted medium sand specimens at 30% relative density were  $6.06 \times 10^{-6}$  cm/s,  $9.21 \times 10^{-6}$  cm/s,  $9.58 \times 10^{-6}$  cm/s and  $9.76 \times 10^{-6}$  cm/s at the end of 7<sup>th</sup>, 28<sup>th</sup>, 56<sup>th</sup> and 150<sup>th</sup> day curing periods respectively. Increase in the permeability of sodium silicate grouted sand samples with time was due to syneresis of grout gel matrix of sand specimens.

**CONCLUSION**

Followings were the main conclusions drawn from this experimental study:

- The gel time of grouts were decreased with the increase in sodium silicate content.



**Figure 10.** Variations of permeabilities of grouted sand specimens with fine sand content for 150<sup>th</sup> day.

- The onset viscosity was increased with the increase in silicate content.
- Syneresis of grouts increased as the silicate content increased up to 37% but after that started decreasing as the silicate content increased.
- Different graded sand specimens compacted at 30, 50 and 70 percent relative densities were successfully permeated with all contents of sodium silicate solutions.
- The permeability of different graded sand samples at various relative densities was reduced two to five orders of magnitude by grouting.
- Increase in fine content decreased the permeabilities of injected sand samples.
- Increase in relative density slightly decreased the permeabilities of grouted sand specimens.
- Up to 37% of silicate content, the hydraulic conductivity of injected sand samples increased. After that it decreased with the increase in silicate content.

## ACKNOWLEDGEMENTS

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