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Effect of cement, sodium silicate, kaolinite and water on the viscosity of the grout

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Cement, sometimes with/without other binders, is widely used for the stabilization of soil by injection and grouting methods. Chemical grouts and sodium silicate are commonly used worldwide for stabilizing soft soils. This article presents the effectiveness of using cement, sodium silicate, water and kaolinite on the viscosity of the grout. Different samples with different ratio of grout components were prepared and the changes in the viscosity of grout were evaluated. The results showed that by increasing cement, kaolonite and sodium silicate, the viscosity of grout increased; on the other hand, it decreased with an increase in water. The effect of kaolinite on the grout viscosity was investigated is more than that with cement and the behaviour of grout without sodium silicate was observed to be similar to the thixotropy fluid behaviour, and it was similar to rheopexy fluid behaviour with high amount of sodium silicate.

Key words: Cement-sodium silicate system grout, viscosity, thixotropic fluids, rheopexy fluids.

INTRODUCTION

Grouting and chemical grouting technologies have grown over the last few decades. For many years, the term chemical grout was synonymous with sodium silicate grouts, but in the last three decades, many different chemical compounds have been produced which provide a wide selection for grouts (CIRIA, 2000; Shroff and Shah, 1999; Wallner, 1976). Sodium silicates have been developed into a variety of different grout systems. These systems contain sodium silicate and a reactor/accelerator (e.g., calcium chloride), which is compatible with cement, to get strong bonding properties in a two-compound system. This grout system that is, sodium silicate and the reactant solution with cement can be injected separately in two steps. The two compound system has also been used below a water table and produces a high-strength. permanent grout if not allowed to dry out (Krizek and Pepper, 2004; Karol, 2003; Shroff and Shah, 1999; Taylor, 1997; USACE, 1995; Clarke, 1984).

The viscosity and the rheology of grouts is a way of describing its properties without paying any attention to

whether it is a homogenous grout or a mixture of grains in a grout (Eklund, 2005; Birld et al., 1987). It is normally applied to fluid materials (or materials that exhibit a time dependent response to stress). It can also be defined as the study of the flow of the grout flows, before the setting point is reached. This is crucial due to the fact that the grout must be placed by some kind of mechanical process like, pumping into the prepared forms. Cement grout based rheology is characterized by at least two parameters; yield stress and plastic viscosity. In a similar way, an elastic solid is characterized by two parameters; Young's modulus and Poisson's ratio (Bentz et al., 2006; Amadei and Savage, 2001).

Hansson (1993) stated that the rheological behavior of a cement-based grout is difficult to define because of the concentration and characteristics of the particles as well as the suspension medium. The rheological behavior and viscosity are influenced by the chemical reactions in progress during the hydration of the cement and the thixotropy is dominant at short cycle times.

Newtonian and non-Newtonian fluids

The Newtonian fluids show that the relationship between

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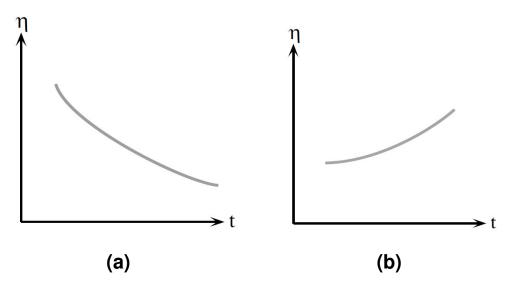


Figure 1(a). Thixotropic fluids behaviour, and **(b)** Rheopexy fluids behaviour. This article presents the results of the effect of different additives on the viscosity of the grout.

shear stress (τ) and shear rate (γ ') is a straight line. Brookfield Engineering Labs Inc. (2010) stated that, in practice, at a given temperature, the viscosity of a Newtonian fluid will remain constant regardless of which viscometer model, spindle or speed is used to measure it.

A non-Newtonian fluid is a fluid for which the relationship $^{\gamma}$ is not a constant. In other words, when the shear rate is varied, the shear stress does not vary in the same proportion (or even necessarily in the same direction). Chhabra and Richardson (2008) and Syrjälä, (1996) defined the non-Newtonian fluid as one whose flow curve (shear stress versus shear rate) is either nonlinear or does not pass through the origin, that is where the apparent viscosity (ratio of shear stress to shear rate) is not constant at a given temperature and pressure but is dependent on the flow conditions such as flow geometry, shear rate, etc.

Thixotropic fluids and rheopexy fluids

Nguyen and Uhlherr (1983) explained thixotropic fluids as follow: a material is said to exhibit thixotropy if, when it is sheared at a constant rate, its apparent viscosity (or the corresponding shear stress) decreases with the time of shearing, as can be seen in Figure 1(a), such as red mud suspension containing solids. Struble and Ji (2001) observed the behavior of cement grout to be similar to thixotropic fluids. The rheological parameters of grouts influence the course of the grouting. Eriksson (1998) illustrated different examples showing how the viscosity and yield value changed the spreading of the grout in a defined geometry. Hansson (1993) stated that the rheology changes in grouts depend on the w/c ratio and the specific surface of the grout.

The fluids for which their apparent viscosities increase with time of shearing are said to display rheopexy or negative thixotropy (Figures 1b). In a rheopectic fluid, the structure builds up by shear and breaks down when the material is at rest. In other words, in contrast to thixotropic fluids, the external shear encourages the build-up of structure in this case (Singh and Chhabra, 2009; Chhabra and Richardson, 2008; Tanner, 2000; Keller and Keller, 1990; Griskey et al., 1985; Pradipasena and Rha, 1977; Steg and Katz, 1965; Metzner and Whitlock, 1958; Freundlich and Juliusburger, 1935).

MATERIALS AND METHODS

Evaluations of tests

Materials

Cement used as the first binding agent, was obtained from Anuza Enterprise Company, Serdang, Malaysia. The composition of cement, as reported by the manufacturer, was CaO (65.6%), SiO₂ (21%), Al₂O₃ (5.3%), Fe₂O₃ (3.3%), SO₃ (2.7%), MgO (1.1%), Na₂O (1%), and the loss on ignition was 0.9%. Hydrous sodium silicate, a syrupy liquid, was used as the second binding agent. It consisted of SiO₂ (28.7%), Na₂O (8.9%) and the silica ratio (SiO₂/Na₂O) was 3.22. The density was 1.38 Mg/m³ and pH was 11.3, as reported by the manufacture.

Calcium chloride (CaCl₂), an anhydrous powder, was used as a reactor/accelerator. It behaves as a typical ionic halide and is solid at room temperature. It can serve as a source of calcium ions in a solution unlike many other calcium compounds, calcium chloride is soluble. Sodium silicate and calcium chloride were obtained from Merck Sdn. Bhd., Petaling Jaya, and Bendosen Company, Selangor, Malaysia respectively. The Kaolinite [Al₂Si₂O₅(OH)₄] structure is made up of silicate sheets (Si₂O₅) bonded to aluminum oxide/hydroxide layers [Al₂(OH)₄] called gibbsite layers. It consisted predominantly of SiO₂ (45.8%) and Al₂O₃ (39.55%), as detailed in the properties sheet supplied by the manufacturer. It was sourced

Table 1. Grout formulae with their notations and group numbers.

Grout no.	Na (%)	Ce (%)	K (%)	W (%)
1	0	20	20	30
2	0	30	20	30
3	0	20	30	30
4	0	30	30	30
5	0	20	20	40
6	0	30	20	40
7	0	20	30	40
8	0	30	30	40
9	30	20	20	40
10	30	30	20	40
11	30	20	30	40
12	30	30	30	40
13	0.5	20	20	40
14	0.5	30	20	40
15	0.5	20	30	40
16	0.5	30	30	40
17	1	20	20	40
18	1	30	20	40
19	1	20	30	40
20	1	30	30	40

Na: Sodium silicate, Ce: cement, K: kaolinite, and W: water.

from Kaolin Sdn. Bhd. factory in Puchong, Malaysia.

Sample preparation and test procedure

In order to investigate the viscosity of cement-sodium silicate grout with calcium chloride and kaolinite, different quantities of calcium chloride (0.5 mol/L), kaolinite, sodium silicate, cement and water were admixed together in the ratio as shown in Table 1. For preparing the samples, the optimum amount of kaolinite, calcium chloride, cement and sodium silicate were selected based on USACE (1995) and CIRIA (2000), according to the weight of the wet soil. The weight of wet soil was 100 g and the required volume of the grout was approximately 7 ml. The compounds were mixed with different ratios of water using a household mixer to achieve homogeneity. The temperature in the laboratory was maintained constant at $22 \pm 2\,^{\circ}\mathrm{C}$, as the viscosity of grout can change with a change in temperature.

In this research article, the viscosity of cement-sodium silicate system grout with kaolinite was investigated by Brookfield viscometer (DV-II + Pro). The defined geometry system has been provided by a small sample adapter (Figures 2b and c) for accurate viscosity measurements at precise shear rates. It consists of a cylindrical sample chamber (with volume between 2 - 16 mL) and spindle, which are attached to all standard Brookfield Viscometers/Rheometers. In comparison with other spindles, it can measure the shear stresses with different shear rates and by combination of this accessory and Brookfield rheocalc32 software, the mathematical model of grout behavior can be defined. By using a small sample adapter (model SC4-13RP), the viscosity of fluid can be measured in the range of 1.5 - 30 K, shear rate (s⁻¹) 1.32 RPM, and sample volume to be used is 6.7 mL (Brookfield Engineering Labs Inc., 2008). A pictorial view of the viscometer (DV-II + Pro), Small sample adapter and a detailed schematic section of the small sample adapter is presented in Figure 2.

This viscometer, shown in Figures 2a and b, in conjunction with UL adapter, can measure the viscosity as low as 1 cP. It had the facility for selecting 54 different speeds of the spindle (Figure 2c). The data can be displayed digitally and it is equipped to measure the torque, shear rate and many other parameters. The Small Sample Adapter (SSA), shown in Figure 2(d), consisting of a cylindrical sample chamber and spindle, provides a defined geometry system for accurate viscosity measurements of small sample volumes in the order of 2 to 16 mL at precise shear rates. The SSA's rheologically correct cylindrical geometry provides extremely accurate viscosity measurements and shear rate determinations. This model can automatically control and collect data with Rheocalc32 and a dedicated computer. Rheocalc32 can analyze data, generate multiple plot overlays, print tabular data, run math models and perform other time-saving routines. Data can be saved in the program or exported to excel.

RESULTS

Effect of cement on viscosity of grout

The effects of different ratios of cement (20 and 30% by the weight of wet soil) on the variation of viscosity of grouts with times were investigated. The influence of cement on the viscosity of grout numbers 1 and 2 are presented in Figures 3a and b. According to Table 1, all the components in the grout numbers 1 and 2 are the same, except the cement content, that was increased from 20 to 30%. The variation of viscosity with time for grout numbers 1 and 2 is presented in Figure 3. It was observed that the viscosity of the grout No. 1, with 20% cement, was 24 cP after 100 s of mixing. It increased to

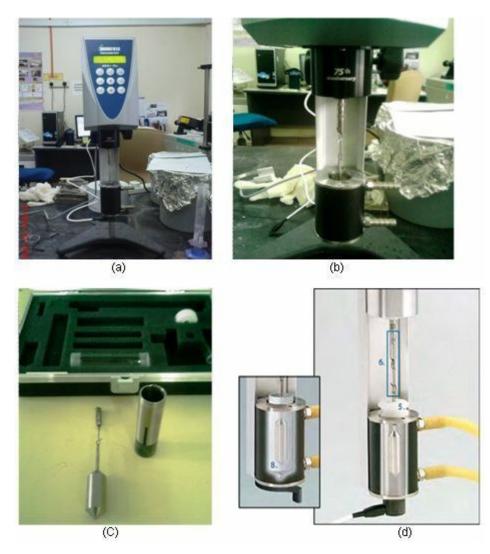


Figure 2. (a) Viscometer (DV-II+Pro), (b) Close-up of small sample adapter, (c) Spindle, and (d) Cross-sectional view of small sample adapter (after, Brookfield Engineering Labs Inc., 2010b).

48 cP when the cement content was increased to 30% (grout No. 2). A similar increase in the viscosity, with an increase in the cement content, was observed with all other grouts mentioned in Table 1, with the same components but different cement content. This behavior of grout is similar to the behavior of the thixotropic fluids.

Effect of kaolinite on viscosity of grout

The effect of kaolinite on the viscosity of the grouts was investigated by preparing samples with different kaolinite content (Table 1) and some typical results are presented in Figures 3(a) and 4. The grout numbers 1 and 3 consist of different amounts of kaolinite (20 and 30% by the weight of wet soil), while the amount of other additives was constant.

The viscosity of the samples with grout numbers 1 and

3 was observed to increase with an increase in the kaolinite content after mixing. For grout No. 1, with 20% kaolinite, the viscosity was 24 cP after 100 s (Figure 3a). When the kaolinite content was increased to 30% (grout No. 3), the viscosity increased to 60 cP after 100 s of mixing (Figure 4).

Effect of water on viscosity of grout

The effect of water on the viscosity of the grout was studied by preparing samples with different amount of water (30 and 40% by the weight of wet soil). Its influence on the viscosity of the grout with an increase in time is presented in Figures 4 and 5 for the grout numbers 3 and 7, respectively. This observation of a decrease in viscosity with an increase in the water content was also observed for the different grouts mentioned in Table 1.

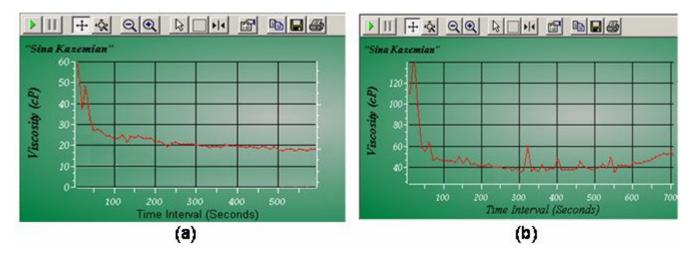


Figure 3. Viscosity of grout versus time (a) grout number 1 (b) grout number 2.

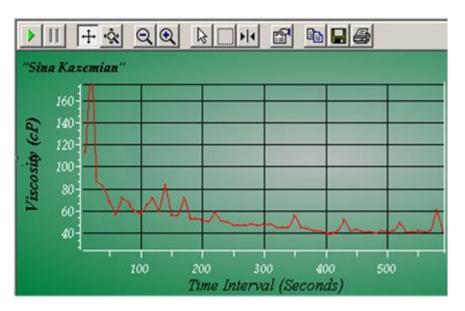


Figure 4. Viscosity of grout number 3 versus time.

The viscosity of the grout No. 3 (30% water) was 60 cP after 100 s of mixing. When the water content was increased to 40% (grout No. 5), the viscosity reduced to 28 cP after 100 s of mixing. A similar reduction was observed with other grouts also upon an increase in the water content.

Effect of sodium silicate on viscosity of grout

The effect of sodium silicate on the viscosity of the samples were investigated by adding sodium silicate (0, 0.5, 1 and 30%) by the weight of wet soil (Table 1) and some typical results are presented in Figure 6. The effect of sodium silicate on the viscosity of grout was studied

under three different categories: (i) without sodium silicate (Figures 3, 4 and 5), (ii) with high amount (30%) of sodium silicate (grout No. 10, Figure 6a), (iii) with low amount (1%) of sodium silicate (grout No. 18, Figure 6b).

For grout No. 10 (30% sodium silicate, Figure 6a), the viscosity was 17 cP after 20 s of mixing. It increased gradually to 23 cP after 40 s, but rose sharply to 120 cP after 100 s. However, for grout No. 18 (1% sodium silicate, Figure 6b), the viscosity was 65 cP after 100 s and 58 cP after 200 s. Furthermore, by using a low amount of sodium silicate in the grout, the behavior of viscosity vacillated. It decreased from 60 to 40 cP in the initial time after mixing (around 350 s) and then it started to increase again. This trend was observed for most of the grouts with low amount of sodium silicate.

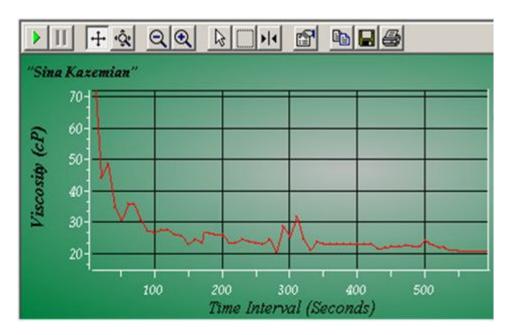
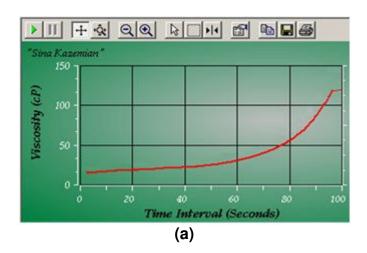


Figure 5. Viscosity versus time of grout number 7.



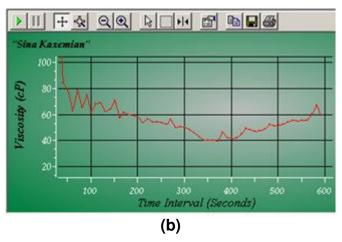


Figure 6. Viscosity of grout versus time: (a) grout number 10 and (b) grout number 18.

DISCUSSION

Effect of cement on viscosity of grout

It was observed that the viscosity of the grouts increased with an increase in the cement content. This behavior was observed with all the grouts mentioned in Table 1, with the same components but different cement content. This behavior of grout is similar to the behavior of the thixotropic fluids.

It should be mentioned that when water comes in contact with cement, three phenomenon take place; (i) cement reacts with water called hydration, (ii) pozzolanic reactions between Ca(OH)2 from burnt cement and pozzolanic minerals in the soil and (iii) ion exchange between calcium ions (from cement and additives) with ions present in the mineral part grout. The increase in the grout viscosity is because of the hydration reaction between cement and water. With an increase in the cement content, there is an increase in the hydration reaction leading to absorption and higher utilization of water in the mixture; thus increasing the viscosity of the grout. As mentioned earlier, the amount of kaolinite and mineral parts in the grout were constant, but due to an increase in the cement content, the viscosity of the grout increased due to the dominance of the second and the third reaction (pozzolanic reaction and ion exchange).

Effect of kaolinite on viscosity of grout

The viscosity of the samples with grout numbers 1 and 3 was observed to increase with an increase in the kaolinite

content after mixing. For, grout No. 1, with 20% kaolinite, the viscosity was 24 cP after 100 s (Figure 3a). When the kaolinite content was increased to 30% (grout No. 3), the viscosity increased 60 cP after 100 s of mixing (Figure 4).

The trend of variation in viscosity upon increasing the kaolinite content for the samples with other grouts was similar to trend for the grout numbers 1 and 3. The effect of the kaolinite in the samples is the reactions among kaolinite, cement (pozzolanic reactions) and sodium silicate. These reactions, upon increasing the kaolinite content from 20 to 30%, lead to an increase in the shear strength and a corresponding decrease in the moisture content. By comparing the viscosity of grouts numbers 2 and 3, it was obvious that the effect of kaolinite on the viscosity of the samples is more than that with cement. It is because of the difference in the density of kaolinite and cement. Kaolinite has lower density than cement and hence a higher volume of kaolinite for the same weight. Thus, the surface area of kaolinite particles is more than that of cement particles. The specific surface area of kaolinite reported by many researchers (Castellini et al., 2008; Zbik and Smart, 1998) is in the range of 10 - 29 m²/g; whereas, the specific surface area of cement is reported to be in the range of 0.8 - 1.0 m²/g (Bye, 1996). Hence, it is obvious that the kaolinite particle can absorb higher amount of water compared with the cement particles.

Effect of water on viscosity of grout

The effect of water on the viscosity of the grout was studied by preparing samples with different amount of water (30 and 40% by the weight of wet soil). Its influence on the viscosity of the grout with an increase in time is presented in Figures 4 and 5 for the grout numbers 3 and 5 respectively. As mentioned earlier, a decrease in viscosity was observed with an increase in water content. Similar observations were made for the different grouts mentioned in Table 1.

The viscosity of the grout number 3 (30% water) was 60 cP after 100 s of mixing. When the water content was increased to 40% (grout number 5), the viscosity reduced to 28 cP after 100 s of mixing. A similar reduction was observed with other grouts also upon an increase in the water content. As mentioned earlier, when water comes in contact with cement, three different reactions take place; hydration, pozollanic and ion exchange. In this condition, on adding more water to the grout, while other components of the grout are constant, it causes to dilute the mixture and decreases the intensity of the reactions and hence a decrease in the viscosity.

Effect of sodium silicate on viscosity of grout

The effect of sodium silicate on the viscosity of the

samples was investigated by adding sodium silicate (0, 0.5, 1 and 30%) by the weight of wet soil (Table 1). As mentioned earlier, the effect of sodium silicate on the viscosity of grout was studied under three different categories; without sodium silicate, high sodium silicate content and low sodium silicate content.

The behavior of grouts without sodium silicate are similar to the behavior of grouts numbers 2, 3, and 5 as shown in Figures 3, 4, and 5 respectively. This has been already earlier and the results agree well with the findings of Struble and Ji (2001). Further, the behavior of cement grout is the same as that of the thixotropic fluids. The behaviour of grout number 10 (Figure 6a) with 30% sodium silicate was observed to be similar to those of rheopexy fluids.

As mentioned earlier, for grout number 10 (30% sodium silicate), the viscosity was observed to increase up to 100 seconds. However, for grout No. 18 (1% sodium silicate), the viscosity was observed to decrease after 200 seconds. On using a low +amount of sodium silicate in the grout, the behavior of viscosity was observed to vacillate. This trend was observed for most of the grouts with low amount of sodium silicate.

These findings showed that, the behavior of grouts with high sodium silicate and with no sodium silicate is opposite (thixotropic fluids and rheopexy fluids behavior). Cement can intensify the reaction with sodium silicate and leads to a large increase in the viscosity of the grout in a very short time. This is because of the reaction between cement and sodium silicate, in the presence of calcium chloride, which causes the clinker minerals contained in the confection which are intensively hydrated and the OH ions pass into the solution to be consumed there for the depolymerization and hydrolysis of the silicate anions of the additive and for an increase in the pH value of the liquid phase (Aborin et al., 2001). Calcium unlike many other calcium compounds is soluble. This property can be useful for displacing ions from solution. During this period, the hydrated calcium silicates are formed, via the precipitation of silicate ions of the additive and also via the release of silicate and aluminate ions from the clinker which cause decrease viscosity of grout. Aborin et al. (2001), Karol (2003) and Yonekura and Kaga (1992), who have reported that if alkaline solution with sodium silicate (sodium silicate solutions are alkaline) concentration above 1 or 2% by volume is neutralized by reactants like acid salts (calcium chloride) or certain acids, a dilute sodium silicate solution will aggregate to form a gel after a time interval.

Based on trend of the variation in the viscosity of the grout with time, due to an increase in sodium silicate, it can be concluded that the problem of bleeding in cement grout can be controlled by adding sodium silicate to the cement grout. Furthermore, it is not possible to use one compound system for grouting (cement-sodium silicate system grout) as there is a very rapid onset of reaction among the grout components; hence two compound

system grouts should be used for injection. This finding also agrees well with the findings of Clarke (1984), Karol (2003) Shroff and Shah (1999) and USACE (1995).

Conclusions

This study was carried out to investigate the effect of the components of the cement-sodium silicate grout and kaolinite on the viscosity of the grout. The change in the viscosity of the samples with time was studied by Brookfield viscosity meter. The following conclusions are drawn based on this study:

- (1) An increase in the amount of kaolinite will cause a decrease in the viscosity of the grout. It is because of the reactions between the kaolinite, cement (pozzolanic reactions) and sodium silicate. The effect of kaolinite on the viscosity of the grout was higher than that of cement because of the density of kaolinite is less than the density of cement. Hence, the surface area of the particles of kaolinite is more than that of cement, for the same weight of the materials.
- (2) The viscosity of grout decreases with increasing amount of water. This is due to the fact that the water reduces the intensity of reactions and causes the grout to be diluted.
- (3) The effect of cement and sodium silicate on viscosity of grout showed that by increasing the ratio of cement and sodium silicate on the samples the viscosity increased. This happens since the hydration and pozzolanic reactions of cement, ion exchange between calcium ions with ions present in the mineral part of grout, and the rapid reaction between cement, sodium silicate, and calcium chloride forms colloids which polymerizes further to form a gel that binds soil particles together.
- (4) By increasing sodium silicate from 0 to 30%, the viscosity curve changed to three different shapes. It is not possible to use one compound system for cement grout with high amount of sodium silicate, due to very rapid onset of reactions among the grout components.

Hence, two compound system grouts should be used for injecting sodium silicate system grout. The bleeding problem of cement grout can be avoided by using low ratio of sodium silicate with the cement grout. At the same time, there will not be much reduction in the viscosity of the cement grout.

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