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Treatment of Medium- to Coarse-Grained Sands by Fine-Grained Portland Cement (FGPC) as an Alternative Grouting Material to Silicate-Ester Grouts

ABSTRACT: Due to the inability of Ordinary Portland Cement (OPC) grouts to permeate such soil formations as fine- to medium-grained and/or medium- to coarse-grained sands and the problems associated with permanence and toxicity of chemical grouts, advanced studies have shown that fine grained portland cement (FGPC) based grouts may be used to overcome the difficulties mentioned above and hence, an opening in the market has appeared for the manufacture of very fine-grained cements. In this context, comparative laboratory studies were conducted on commercially available OPC, FGPC, and silicate-ester grouts, and it has been found that FGPC has better flow properties and bleed characteristics than OPC. Furthermore, its permeation into medium- to coarse-grained sand is as effective as silicate-ester grout and the strength of the sand gained by the injection of FGPC is higher than that of silicate-ester grouted sand.

KEYWORDS: fine-grained portland cement, silicate-ester, treatment, grouting, strength, permeability

Introduction

Grouting of soils is often done to reduce the permeability and/or improve the mechanical properties of a formation (soil and rock). Grouts are generally classified as either suspensions or solution grouts. Suspension grouts are prepared with ordinary portland cement or other cements, clays, or clay-cement mixtures and successfully injected into gravel and, to some extent, coarse-grained sands, but the permeation of these grouts into fine- and medium-grained sand is difficult and often impossible.

Chemical grouts are solution grouts made up of two or more chemicals, which react to form a gel. The most commonly used grouts are based on sodium silicates, phenoplasts, acrylamides, lignins, or resins. Chemical grouts are generally more expensive than cement-based grouts, but within chemical grouts, silicate-based grouts are usually the least expensive. Chemical grouts are used to treat soils with finer granulometry; however, many chemical grouts pose problems such as permanence and toxicity. For this reason, the use of fine-cementitious grouts has been recommended but the information about the performance of such grouts is not sufficient. Therefore, this laboratory investigation focuses on the rheological properties of a fine-grained portland cement grout, the penetrability of this grout into medium to coarse-grained sand, and its effectiveness in terms of strength and permeability of the grouted sand in comparison with OPC and silicate-ester grouts.

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Physical and Chemical Properties of Cement-Based Grouting Materials

Fine-Grained Portland Cement and OPC

Adding a small amount of calcium sulfate to cement clinker produces anhydrous ordinary portland cement. The resulting portland cement is a heterogeneous mixture of several minerals produced by high temperature reactions between the chemicals summarized in Table 1 (Mehta, K.P., 1986).

The fine-grained Portland cement (FGPC) used in this study was Microcem H900, manufactured by Blue Circle Industry plc in England in a 'Ball Mill' works. OPC is ground into finer particles and the smallest particles are separated from the resultant powder by introducing it to a cyclone of air created in a confined space. A range of smaller particles separate in the cyclone fall to the bottom, and are then collected for use as a fine-grained cement (De paoli, B. et al., 1992).

The grain size distributions of the FGPC and OPC were determined ultrasonically in water using a Malvern instrument and the results are shown in Fig. 1. Grain sizes of the FGPC range in size from 2–40 μm, whereas 80% of the OPC grain sizes are in between 10 μm and 100 μm. Furthermore, the specific surface area of FGPC and OPC are about 7 700 cm^2/g and 4000 cm^2/g, respectively. Since the FGPC is finer than OPC, its permeation capacity should be higher than that of OPC.

Silicate-Based Grouting Materials

Sodium Silicate and Hardener (Ester)

The sodium silicate employed is manufactured by Imperial Chemical Industry, England, and is marketed M75. Table 2 shows some of properties of M75. A hardener is mixed with the sodium silicate solution as a reagent to form a silica gel. The hardener used in this study is one of the Hardener 600 Series that is manufactured by Rhone Poulenc, France. The Hardener 600 Series is an ester that is commercially graded by the letters B, C, D, and E, which are used to denote the speed of reaction with the silicate, where B is the most rapid action. Some of the physical properties of Hardener 600B are as shown in Table 3.

Grout Properties of FGPC and OPC Suspensions

To mix grouts having different water/cement ratios, the quantities of each constituent, i.e., water and cement, were weighed in separate plastic containers using a Predisa 6 000d, 5 kg balance. The water was then transferred into a larger plastic drum and the cement was introduced gradually, while operating an electric drill with whisk attachment at 1 300 rpm immersed within the water. Grouts were agitated for up to 10 min, which has been shown to be sufficient for experimental studies (Banfill, P.F.G., 1981) and then tested for sedimentation, viscosity, and set time to the following tests. This mixing method was carried out to simulate the colloidal mixing equipment commonly used in the grouting industry.

Sedimentation

When using cement grouts to permeate into joints or pores, not only the rheological properties that are important and influence the success of the grouting operation, but also the resistance to sedimentation (bleeding), and the size of the cement particles (Schwarz, L. G. et al, 1992). A cement grout is said to be stable if the sedimentation due to gravity is zero. A commonly used test for bleeding is ASTM C 940. The volume of clear water on top of a 1000 mL graduated cylinder divided by the original grout volume must be less than 5% after 2 h (Deere et al., 1985). A stable grout free from sedimentation or bleeding is generally considered to be more favorable than an unstable grout in practical grouting. To monitor the bleed capacity of each grout mixture prepared, a sample was taken from the agitator approximately 10 min after mixing and placed into 1 000 mL graduated cylinder. The result of sedimentation tests (up to 24 h) conducted on OPC and FGPC grouts with different water /cement ratios are given in Tables 4 and 5.

Bleed capacity of the OPC and FGPC grout suspensions presented in Tables 4 and 5 shows that the OPC grout has a higher bleed capacity. This is attributed to differences in the grain-size distribution of the two materials. Furthermore, after two h of sedimentation period, the bleeding capacities of the OPC and FGPC grout suspensions with w/c ratios of 0.8–1.2 are 19%, 40%, 1%, and 4% respectively. This indicates that the FGPC is a stable cement grout based on the criterion noted earlier (Deere et al. 1985).

TABLE 1—*Typical oxide analysis of OPC and/or FGPC.*

Oxides	%
SiO_2	20.2
Al_2O_3	5.7
Fe_2O_3	2.6
Mn_2O_3	0.09
P_2O_5	0.05
TiO_2	0.28
CaO	63.50
MgO	1.2
SO_3	3.7
LOI	1.5
K_2O	0.74
Na_2O	0.10
Free Lime	1.8

TABLE 2—*Silicate solution produced by ICI.*

Baume Degree	SiO_2/Na_2O (by weight)	Viscosity (cP) at 20°C	SiO_2 (%)	Na_2O (%)	H_20 (%)
39.4	2.9	100	26.8	9.2	64

TABLE 3—*Physical properties of FGPC.*

Melting point	< -30°C
Fire point	118°C
Density at 20°C	1.09
Viscosity at 20°C	5 Cp

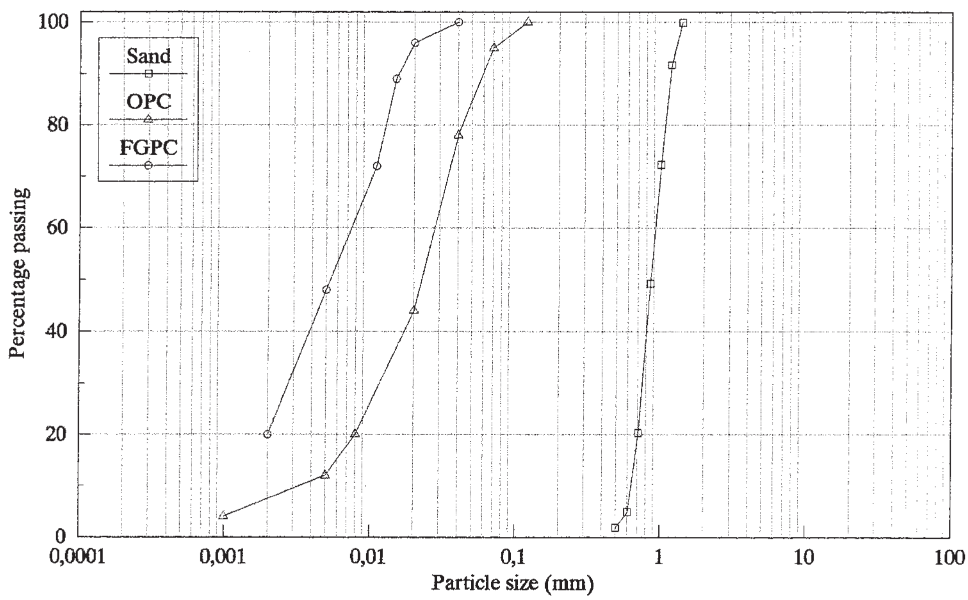

FIG. 1—*Particle size distribution curves.*

TABLE 4—*Bleed capacity results for OPC.*

W/C	Wet Density (Mg/m3)	Mix Temp. (°C)	Ambient Temp. (°C)	Bleed Capacity (%) 1 h	2 h	3 h	24 h
0.8	1.60	20	Approx. 17 °C	17	19	19	18
1	1.51	22		32	32	32	31
1.2	1.45	22		40	40	41	40
1.4	1.40	23		45	44	45	45

TABLE 5—*Bleed capacity results for FGPC.*

W/C	Wet Density (Mg/m3)	Mix Temp. (°C)	Ambient Temp. (°C)	Bleed Capacity (%) 1 h	2 h	3 h	24 h
0.8	1.61	1.61	Approx. 17 °C	1	1	1	0
1	1.52	1.52		1	2	2	2
1.2	1.45	1.45		4	4	6	10
1.4	1.38	1.38		6	12	15	15

Viscosity

Viscosity tests were performed using Haake Rotovisco RV20 viscometer to determine plastic viscosity and yield stress of the FGPC and the OPC grout suspensions. Immediately after mixing was completed, the grout was poured into the container and testing was started. Measured values are given in Table 6.

The results in Table 6 indicate that there is no noticeable difference in the viscosity of the OPC and the FGPC grout suspensions, but as the particle's size becomes smaller the yield stresses of the suspensions increases. This is due to the rate of hydration increase caused by the increase in specific surface area, which in turn results in resistance between particles against shearing (Littlejohn, G. S., 1982).

TABLE 6—*Viscosity of FGPC and OPC grout suspensions (at ambient temp. of 20°C).*

W/C	Viscosity (Pa.s.) FGPC	OPC	Yield Stress (Pa) FGPC	OPC
0.8	0.03	0.02	13	2
1	0.02	0.02	4.7	1
1.2	0.02	0.02	1.6	1
1.4	0.02	0.01	1	1

Set Time

The setting process may be considered as having two stages; an initial stage in which the fluidity of the grout decreases to a level at which it is no longer pumpable and a second stage in which the grout hardens and attains significant strength termed 'final set' (Schwarz et al. 1992). For a successful grouting operation, it is necessary to determine the initial time of setting of suspension grouts. To determine the time of setting, tests were conducted in accordance with ASTM Test Method for Time of Setting or Hydraulic Cement by Vicat Needle (C191). The time of setting (the limit of pumpability) of the OPC and the FGPC grouts are given in Table 7.

As seen in Table 7, an increase in the water-cement ratio increases the set time of all the grout mixtures. The FGPC grout mixtures exhibited much greater increases in time of setting with increases of the water-cement ratio then the OPC grout mixtures. Furthermore, the time of setting of cement grouts is influenced by particle size. That is to say, as the specific surface area increases hydration rate increases thus reducing the set time (Banfill, P.F.G., 1981).

TABLE 7—*Set time data for OPC and FGPC grouts (at room temperature of 17～20 °C).*

Grout Type	W/C	Mixing Time (min.)	Initial Set Time (min.)
OPC	0.8	10	710
	1.0	10	720
	1.2	10	760
	1.4	10	770
FGPC	0.8	10	210
	1.0	10	420
	1.2	10	600
	1.4	10	765

TABLE 8—*Silicate-Ester grout mixtures.*

Silicate (%)	Hardener (ester) (%)	Water (%)
50	7	43
60	7	33

Silicate-Ester Solution

The desired chemical grout solutions were made by mixing the pre-measured quantities of distilled water, sodium silicate (M75), and ester hardener (600B) in a glass jar sealed with a rubber stopper. The glass jars were then fixed into a laboratory mixer and mixed thoroughly at a speed of 1 Hz for approximately two min. The selection of chemical grout mixtures frequently used in practice was adopted in this research and are shown in Table 8.

Syneresis

Although most of the silicate formulations are considered permanent materials, the end product is subject to syneresis, which often tends to cause doubt about permanence. Newly made silicate-ester grout gel, preserved in an airtight bottle for three years, exuded water and shrank between 3 and 10% of total volume of the original grout mixture, which is called syneresis and occurs at a decreasing rate with time (Mollamahmutoğlu, 1992).

Viscosity

A Rotovisco or Haake rotational viscometer was used to determine the viscosity of the silicate-ester grouts at 15–30 min. The results for 50% and 60% silicate content with 7% Hardener (ester) are shown in Table 9.

Set (Gel) Time

A solution of sodium silicate mixed with ester hardener will undergo a slow saponification producing an open-chain diacid. After a certain elapsed time, the diacid neutralizes the sodium silicate and forms a silica gel. The rate of this reaction depends on the quantity of ester hardener that is necessary to neutralise the sodium silicate. These properties can be controlled by varying the ratio of silicate to the hardener. Gel times of the chemical grout solutions under consideration were obtained at 20°C and are shown in Table 10.

TABLE 9—*Viscosity of Silicate-Ester solutions (at ambient temp. of 20 °C).*

Silicate (%)	Hardener (ester) (%)	Water (%)	Viscosity (Pa.s.)
50	7	43	0.007 ~ 0.008
60	7	33	0.009 ~ 0.013

TABLE 10—*Gel time of Silicate-Ester solutions (at ambient temp. of 20 °C).*

Silicate (%)	Hardener (ester) (%)	Water (%)	Gel Time (min.)
50	7	43	46
60	7	33	55

Sand Specimen Preparation and Grouting

Sand specimens were prepared using a clindrical longitudinally split steel moulds, 38 mm in diameter and 300 mm long. The inside face of each mould was lightly greased with petroleum jelly to prevent the grouted samples from sticking to the side of the mould, as well as facilitating the removal of samples during demolding. The face of the flange of one half mould was coated with silicon rubber sealant to shut off leakage through the flanges when the two halves were joined. After assembling, the split mould was clamped between a top and bottom end plate. Rubber gaskets were employed at the interfaces between the mould and the end plates as a precaution against leakage. The whole assembly was then positioned vertically and filled with water to the top of the mould. Fine gravel, passing 7 mm sieve and retained on a 6 mm sieve, was placed at the bottom of the mould to stop sand from blocking the connecting lines and to distribute the grouts uniformly to the bottom of the sand specimen. A total of 480 g of sand was poured into a narrow-necked volumetric flask through a funnel and the flask was then topped up with water. The sand prepared in this way was then tipped into the mould through the opening in the top plate, light tamping of the sides of the mould was carried out to facilitate sand deposition. Another gravel layer was placed at the top of the compacted sand for the same purpose as mentioned earlier, and the top of the mould attached. The sand specimen was connected to the grout inlet and outlet. After mixing the grout solutions or suspensions were poured into the grout chamber, the chamber was sealed, and the grout injection commenced (Fig. 2). The grout chamber has an air-supply line connected to the top plate. The pressure in the grout chamber is controlled by an air regulator placed between the supply source and the grout chamber. The pressure is observed by a pressure gauge fixed to the top plate. The line supplying the grouts to the grouting moulds are situated at the bottom of the chamber and are also used to clean the chamber after each use. The detailed illustration of injection system used can be seen in Fig. 2.

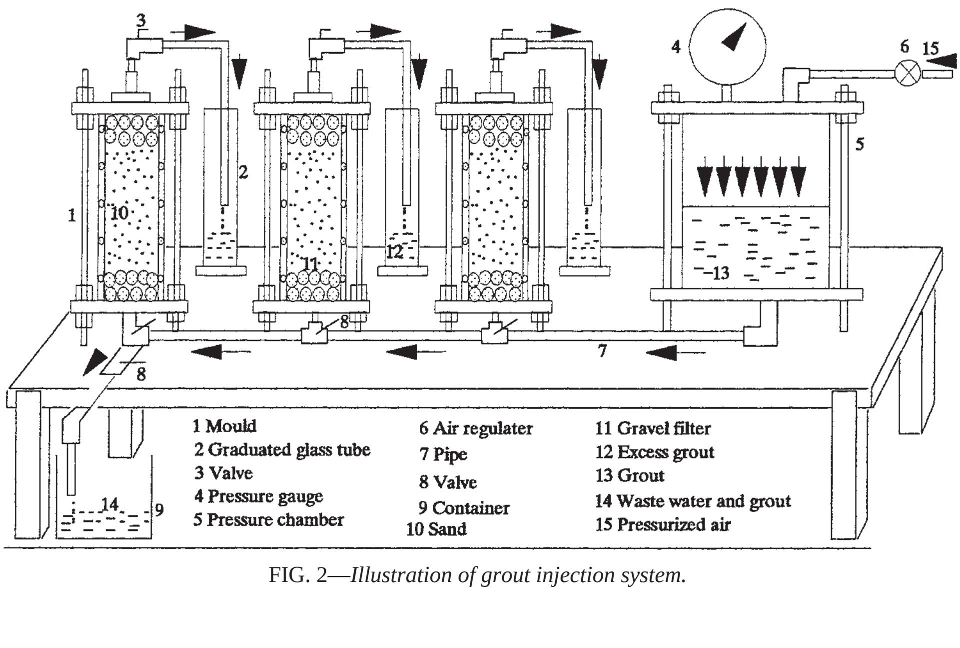

FIG. 2—*Illustration of grout injection system.*

After setting the pressure in the chamber to a fixed value using the pressure regulator, the injection of the grouts into the sand specimens was maintained at a constant flow rate by means of valves at the inlet of each grouting mould. The grouting parameters shown in Table 11 were kept constant throughout the experimental study.

Although grout injection was in progress, graduated cylinders were used to collect pore water as well as excess grout. To ensure that a sufficient quantity of excess grout had been vented, the grouting moulds were then sealed and left standing for the grout to achieve final set.

Permeation of Grouts into Medium-to Coarse-Grained Sand

The sand according to the gradation is shown in Fig. 1 was permeated by OPC, FGPC, and silicate-ester grouts separately. For both cement grouts, a water-cement ratio of 1.2 was adopted as this is commonly used in the grouting industry. The percentages of each component in the silicate-ester grout solutions were also selected as those commonly used in the industry. The sand in the moulds was easily permeated by silicate-ester grouts under a pumping pressure of 20 kPa, but with the FGPC grout, permeation was achieved with 80 kPa of pressure. However, permeation into the same sand was impossible with the OPC using the same w/c ratio. Although the pumping pressure was increased to 250 kPa, very little to no permeation took place. Indeed only ⅕ of the sample height was permeated by OPC grout when hydraulic fracture occurred, ending the test.

Unconfined Compression Tests

Twenty-four hours after injection, the grouting cells were dismantled and samples were cut to the required length for unconfined compressive strength tests. The grouted sand samples were placed inside a longitudinally split plastic casing held together with rubber bands to protect the samples against damage during handling and storage. The protected samples were stored in a 100% humidity room at 20°C until testing.

The grouted samples were tested at a strain rate of 1.52 mm/min. for different time intervals and the unconfined strength values of grouted sand are shown on Table 12. While the unconfined compressive strength of silicate-ester grouted sand specimens remained constant after 24 h, the unconfined compressive strength of the FGPC grouted sand specimens increased with age. As seen from Table 12 the strength gained by the FGPC grout is much higher than that of silicate-ester grout for the grouted sand samples.

Permeability Tests

With a simple arrangement, constant head permeability tests were conducted under a gradient of 20 without removing the grouted sand samples from their moulds. The results of permeability tests are given in Table 13.

Conclusions

The results obtained from this experimental study are follows:

1. The bleed test results for various w/c ratios for the FGPC is considerably lower than that of ordinary Portland cement for the same w/c ratios. Moreover, the FGPC grout suspensions having w/c ratios in between 0.8 and 1.2 are stable grouts as defined by 2-h bleed capacity being less than 5%.
2. As the specific surface area increases, yield stress and the plastic viscosity increase. For instance, while the yield stress and plastic viscosity for OPC grout with w/c ratio of 0.8 are 2 Pa and 0.02 Pa.s., they are 13 Pa and 0.03 Pa.s., respectively, for the same w/c ratio using the FGPC.
3. The set time of cement grouts is influenced by particle size. As the particle size becomes smaller the hydration rate increases as shown by the set time. The set time for OPC grout having w/c ratio of 0.8 is about 12 h but the set time for the FGPC grout with the same w/c ratio is around 7 h.
4. Permeation at 80 kPa grouting pressure into sand having particles in between 0.5 mm and 1.4 mm and a relative density of 70% was achieved by the FGPC grout with a w/c ratio of 1.2. OPC grout with the same w/c ratio, was impossible to permeate even when the grouting pressure was increased to 250 kPa at which time hydraulic fracture induced.
5. Silicate-ester grouts have substantially better flow properties and shorter set times than cement grouts. Furthermore, grouting of sand with 50 and 60% of silicate-ester grouts

TABLE 11—*Some parameters concerned with grouting.*

Relative Density of the Sand (%)	70
Grouting pressure for chemical grouting (kPa)	20
Grouting pressure for cement grouting (kPa)	80
Volume of excess grout	120% of void ratio
Sample preparation	Submerged condition

TABLE 13—*Permeability test results of grouted sand specimens.*

	Coefficient of Permeability, k (cm/sec.)		
Grout Type	7 days	28 days	40 days
W/C = 1.2	No flow	No flow	No flow
50:7:43	No flow	No flow	No flow
60:7:33	No flow	No flow	No flow

TABLE 12—*Unconfined compressive strength of grouted sand*.

		Unconfined compressive strength (kPa)				
Grout Type		1 day	7 days	14 days	28 days	48 days
FGPC (w / c = 1.2)*		2 000	3 100	4 123	4 425	4 428
Silicate-ester	50:7:43*	305	305	305	305	305
	60:7:33*	413	413	413	413	413

* Average values of 3 identical samples for each time intervals.

was accomplished at 20 kPa, indicating a better permeation capacity than the FGPC grout. Permeation into the same sand was possible with the FGPC grout at an increased pressure, but was not possible with the OPC grout at any pressure.

6. Silicate-ester grouts are subject to syneresis, which may affect the performance of the grout in terms of permeability and unconfined compressive strength and may also result in pollution in the ground.
7. Unconfined compressive strength of grouted sand with the FGPC is much higher than that of silicate-ester grouted sand. For example, unconfined compressive strength of the FGPC grouted sand with w/c ratio of 1.2 is 4425 kPa whereas the unconfined compressive strength of 60% silicate-ester grouted sand is 413 kPa at the end of 28 days.
8. Throughout the 40 days of permeability tests there was no flow through the sand specimens injected with both the FGPC grout and the silicate-ester grouts, which indicates that permeation of the FGPC into pores of medium to coarse-grained sand was as effective as silicate-ester grouts.

The main goal of this experimental study was to evaluate and compare the effectiveness of permeation of the FGPC grouts into medium to coarse sand where OPC grouts fail to permeate, and the silicate-ester grouts are questionable in terms of strength and permeability of grouted sand. Additives were not used in the FGPC grouts that may further improve the flow properties and enable the grout suspensions to permeate finer sand formations. Plasticizing admixtures reduce both the yield stress and plastic viscosity of FGPC cement grouts thus improving flow properties (Hakansson et al., 1992). Further work is recommended to evaluate the effectiveness of grouting of the FGPC with additives into fine-to medium-grained sand in terms of strength and permeability.

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