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Research Article

Physical and Mechanical Properties of Injected Granular Soil with Thick Super Plasticized Grouts

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Abstract: The use of super-plasticizers in micro fine or regular cement-based grouts has become of vital importance in advanced professional grouting practices. These super-plasticizers play an important role in the production of more durable grouts with improved rheological characteristics. This report presents a laboratory study of the effect of a new-generation Polycarboxylate Super-plasticizer (PCE) on the inject ability of thick cement grouts into a coarse soil, under different grouting pressures, in comparison to that of a polynaphthalene (SNF) super-plasticizer. Finally, the physical (dry unit weight, porosity and permeability) and mechanical properties (compressive strength, elastic modulus) of grouted specimens with various grouts were examined. The experiments were conducted using different additive dosages with grouts proportioned with a water to cement ratio (w/c) of 0.33, 0.4 and 0.5, respectively. The results showed that PCE super-plasticizer is more effective than the SNF one for the increase of grout inject ability and the improvement of physical and mechanical properties of grouted soil.

Keywords: Cement grouting, strength, super-plasticizers

INTRODUCTION

Cement grouting is the injection under pressure of cement slurry into the voids of a soil or rock mass or into the voids or cavities between these materials and an existing structure. Grouting is widely used in many geotechnical construction projects (Nonveiller, 1989), such as building foundations, dams, tunnels, slope stabilization, pre-stressing anchors, bolts, etc. The main targets of grouting are to improve the strength and durability of the mass and/or to reduce the permeability of the mass (Akbulut and Saglamer, 2003; Welsh, 1998; Saiyouri *et al*., 2008; Anagnostopoulos and Hadjispyrou, 2004; Hsiung, 2009). The strength of a grout is important whenever the purpose of the grouting is to strengthen the ground or an existing concrete structure. The ratio of water to cement (w/c) is the most significant factor that affects the strength of the grout. Consequently, the use of grouts with low w/c ratios necessitates the addition of a super-plasticizer to obtain the appropriate rheological properties of the suspension so that grouts are able to flow sufficiently in boreholes, formation pores and rock joints and ensure grouting effectiveness. Super-plasticizers belong to the most common admixtures used in the production of concrete with high workability, excellent slump retention, high strength and durability, reduced segregation and bleeding, reduced setting shrinkage and creeping, increased resistance to carbonation and chloride ion attack, freezing and thawing durability. In grouting technology, super-plasticizers are widely used as additives in cement grouts due to their potential influence on rheological properties, strength, durability, impermeability and resistance to chemical erosion. Currently, the most widely used super-plasticizers are Sulfonated Naphthalene Formaldehyde (SNF) condensates and a new-generation Polycarboxylate-based dispersants (PCE) composed of comb-like copolymers with grafted chains of polyethylene oxide. The dispersion mechanism of the SNF super-plasticizer occurs because the adsorption of the super-plasticizer anionic polymers can convey a net negative electrical charge to the surface of the cement particles, which induces repelling forces between neighbouring cement particles and causes increased dispersion (Plank and Hirsch, 2007; Kim *et al*., 2000). However, the dispersion mechanism of a PCE super-plasticizer is mainly attributed to steric forces, which are dominant for a copolymer and consist of one main linear chain with lateral carboxylate and ether groups (Uchikawa *et al*., 1997; Yamada *et al*., 2000; Puertas *et al*., 2005). Steric forces provide a higher dispersive action compared to electrostatic forces (Collepardi, 2005). Despite numerous studies have been conducted concerning the use of both dispersion mechanism admixtures for the production of concrete or mortar (Zhang *et al*., 2010; Parra *et al*., 2011; Montes *et al*., 2012; Golaszewski, 2012), there is minimal information

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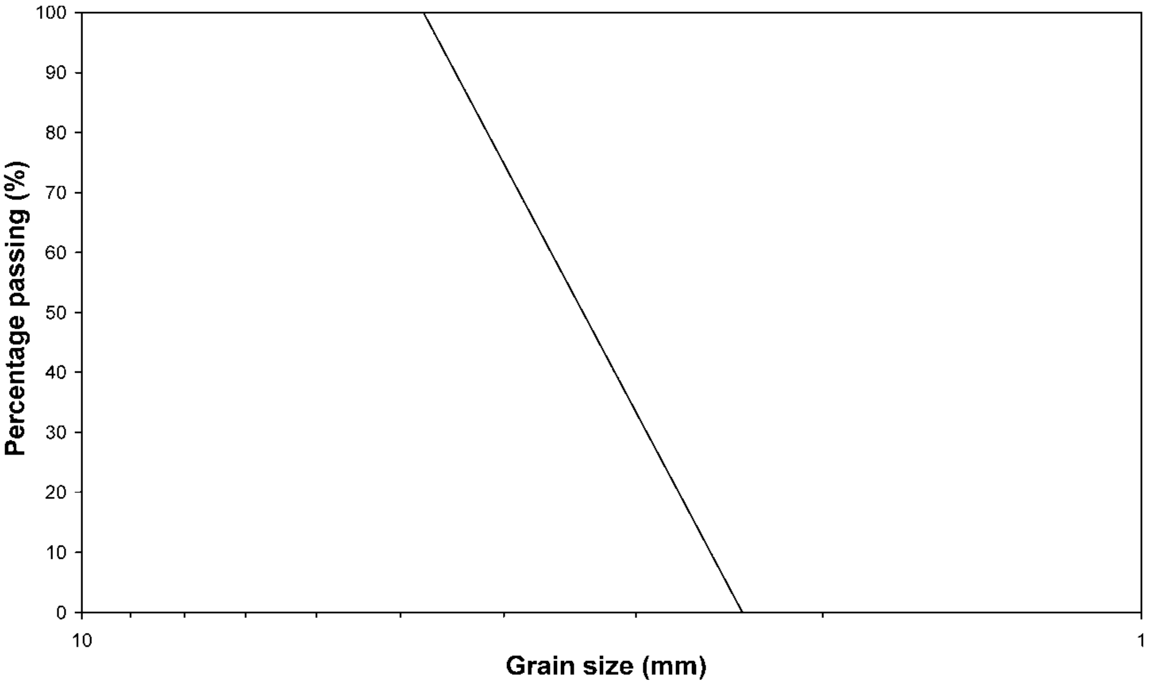

Fig. 1: Particle size distribution of the tested soil

Table 1: Properties of super-plasticizers used in the study

Property	Polycarboxylate ether	Sulfonated naphthalene formaldehyde
Aspect	Slightly yellow	Dark brown
Specific gravity	1.05	1.2
pH	6.3±0.5	6-8
Chloride ion content	Chloride free	Chloride free
Solid content	40%	40%
Molecular mass	44,000 g/mol	16,000 g/mol
Recommended dosage (% by cement weight)	0.6-1.4	0.5-2

available on the effect of the different types of super-plasticizers on various physical and mechanical properties of grouted soils.

The overall goal of this study was to investigate the effect of a new generation PCE dispersant and a SNF one on the inject ability of very thick cement grouts, as well as, their influence on the physical and mechanical properties of a grouted soil, in order to improve the knowledge about the use of such admixtures in cement grouting technology.

MATERIALS USED

Limestone gravel soil with a relative density, D_r, of 43%, was used in the injection tests. The soil was classified as a poorly graded gravel mixture (SP). Its grain size distribution of the soil ranged between 4.76 to 2.38 mm sieves (Fig. 1), with uniformity coefficient of $C_u = 1.6$, dry unit weight of $\gamma_d = 14.3$ kN/m^3, void ratio of e = 0.89, porosity of n = 47 % and permeability coefficient of $k = 4\cdot10^{-2}$ m/sec.

Experiments were carried out using a common type of Portland fly ash-pozzolan cement (CEM II/B-M 42.5 N) according to EN 197-1 (2000) 1 with sulfate resistance properties and a low heat of hydration. It has a specific gravity of 3.15 and a blaine fineness of approximately 4,600 cm^2/g.

A polynaphthalene-based super-plasticizer and a polycarboxylate ether-type super-plasticizer (main chain consisted of a copolymer of methacrylic acid with lateral carboxylate and ether groups, side chain to main chain length ratio of 1) were selected as additives. Both Super-plasticizers are commercial products. Their properties are presented in Table 1.

Laboratory procedure: Grouts with water to cement (w/c) ratios (by weight) of 0.5, 0.4 and 0.33, respectively were used in the injection tests. The dosages of super-plasticizers (% by weight of cement) for all w/c ratios were varied to maintain proper setting times and bleeding values of the grouts, which are proposed by the European Standard EN 12715 (2000) and grouting practices. The final setting time is of great importance to the grouting practice; a short setting time (<4 h) can damage equipment and a long setting time (>24 h) can delay execution of the process and consequently, the grouting efficiency

Table 2: Composition and properties of cement grouts

w/c	Super-plasticizer dosage (%)	Final setting time (h)		Bleeding (%)		Compressive strength after 30 days of curing (MPa)		Elastic modulus after 30 days of curing (GPa)	
		SNF	PCE	SNF	PCE	SNF	PCE	SNF	PCE
0.50	1.0	12.0	13.5	4.1	4.7	40.5	45.5	4.9	5.1
0.40	1.5	14.5	16.5	4.2	4.9	48.5	58.6	5.3	5.6
0.33	2.5	13.0	18.5	4.0	4.8	63.0	68.0	5.7	6.0

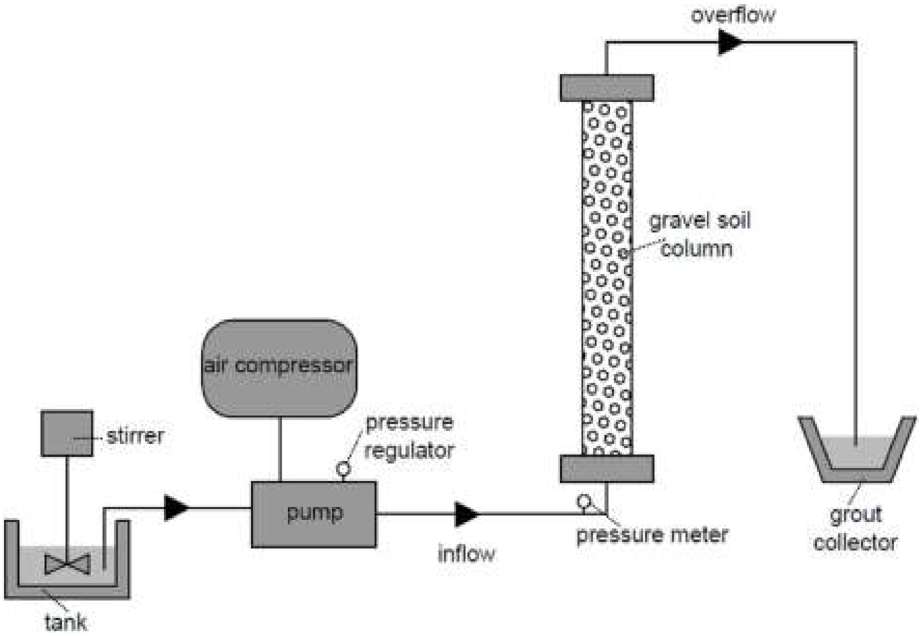

Fig. 2: Testing apparatus for injection experiments

(Perret *et al*., 2000b). Also, according to cement grouting practices, the use of thick stable cement suspensions is compulsory to obtain maximum filling of voids or other spaces that cannot be obtained with the performance of unstable grouts due to their significant bleeding (Houlsby, 1988; Mirza *et al*., 2013). European Standard EN 12715 (2000) characterizes a suspension as stable if it has a bleed capacity of up to 5%. The composition of the grouts and their basic properties are summarized in Table 2. The water content of the Super-plasticizers was accounted for to maintain a constant w/c ratio.

All grouts were prepared using a high speed rotating stirrer to ensure the complete dispersion of cement particles and they were continuously agitated to avoid sedimentation of cement particles during the injection tests. The addition of the super-plasticizer in the grout was performed using the delayed addition method. Particularly, after 5 min of stirring cement and water in the tank and 2 min of static hydration, the appropriate dosage of super-plasticizer was added into the grout. Then, an additional mixing sequence was performed for a total time of at least 2 min. The selection of this method for preparing super-plasticized grout is because the delayed addition of super-plasticizer in cement suspensions significantly enhances the efficacy of its dispersing power in comparison to the direct addition (Fernàdez-Altable and Casanova, 2006; Chiocchio and Polini, 1985; Uchikawa *et al*., 1995; Aiad *et al*., 2002; Flatt and Houst, 2001).

The experimental set-up used for the injections was constructed according to ASTM D 4320-93 (2000) specification. It comprises a mixing tank with a high speed rotating stirrer, an air-operated diaphragm pump, an air compressor, a pressure regulator and pressure meters, plastic moulds 100 mm wide, 1500 mm high and 3 mm thick and the relevant connections (Fig. 2). Before the preparation of the specimens, the inner surface of the molds was lightly lubricated to eliminate sample disturbance upon removal from molds after the end of injection. The weight of gravel soil that fills the mold and gives the required D_r was calculated and this weight of material was poured into the molds. During the filling of the molds, the material was lightly compacted with a rod at different equal layers, until the required unit weight was reached. After placing the specimen at the achievable D_r, the top and bottom end-plates of the mould were clamped using tie-rods.

Injection tests (Table 3) were carried out at constant pressure of 1 and 6 bar, in order to study the efficacy of super-plasticizer on grouting under low or high injection pressure. Injection stopped when no flow

Table 3: Summary and notation of the injection experiments with different grouts

Notation	w/c	SNF (%)	PCE (%)	Injection pressure (bar)
G_1	0.50	1	-	1
G_2	0.50	1	-	6
G_3	0.50	-	1	1
G_4	0.50	-	1	6
G_5	0.40	1.5	-	1
G_6	0.40	1.5	-	6
G_7	0.40	-	1.5	1
G_8	0.40	-	1.5	6
G_9	0.33	2.5	-	1
G_{10}	0.33	2.5	-	6
G_{11}	0.33	-	2.5	1
G_{12}	0.33	-	2.5	6

of grout from the outlet hose of soil column was observed; a consequence of the filtration or clogging mechanism developed inside soil mass during grouting. For the evaluation of the inject ability, the volume of the grout that had passed and collected during the injection was measured, as well as, the initial flow rate of the grout. The quantity of grout flow through the specimen under each applied pressure was measured by using a graduate cylinder. The time for each discharge of grout flow was recorded by a stopwatch for the computation of flow rate.

The grouted specimens were left in the molds for 3 days to develop satisfactory strength and then demoulded and cut into lengths of 20 cm in order to study the physical and mechanical properties of the grouted soil in relation to the distance from injection point. After that, they were sealed in plastic bags and stored in a 95% relative humidity curing room until the day of testing. These cylindrical specimens were used for the estimation of compressive strength and elastic modulus at 30 days of curing according to the specification of ASTM D 4219-02 (2005). It was decided to study the mechanical properties of grouted samples at this age of curing, because previous research has shown that for higher curing periods marginal increase of strength is obtained (Mollamahmutoglu and Yilmaz, 2011; Vipulanandan and Shenoy, 1992). A beta 5 loading machine with a maximum capacity of 3000 KN was used for compression testing at an axial strain of 0.1%/min. The elastic modulus was determined from the linear section of the compressive stress-strain curve.

Specimens of the same size as the one for the mechanical tests, aged 30 days, were used for the evaluation of water permeability by using the method of constant head according to ASTM D 5084-03 (2005) and dry unit weight γ_d according to ASTM C 29/C 29M-91a (1993). Porosity n was calculated according to the method found in Perret *et al*. (2000a). An age of 30 days was chosen for the estimation of the physical parameters. It is considered that beyond this age, the values of physical parameters do not change because the solidification process of the cement hydrates does not influence the density or the volume of the grouted soil and the free water has almost completely evaporated.

Injection experiments were performed on over 100 soil columns to evaluate the aforementioned physical and mechanical parameters of the grouted samples.

RESULTS AND DISCUSSION

The ability of a grout to penetrate porous materials (inject ability) is related with many factors such as w/c ratio, the injection pressure, the physical characteristics of the cement and the physical characteristics of the soil. In the present study, the influence of super-plasticizer type on the inject ability of thick grouts with different w/c ratios and under different injection pressure was examined. The inject ability of the grouts was evaluated based on the initial flow rate and the total volume of grout that had passed from the grouted soil column. In all cases, grouts penetrated the soil column and appeared to have outlet flow. An exception was the grouting with G_9 and G_{11} grout, where outlet flow was not observed and the maximum penetration length was limited, 23 cm for G_9 and 48 cm for G_{11}. This can be attributed to the low injection pressure (low flow velocity) which combined with the high cement content resulted in a very fast blocking of soil voids from the deposition of cement particles inhibiting any further intrusion of the grout. The results of the injection experiments are presented in Table 4. The inject ability of grouts appeared to be directly depended on the w/c ratio, the type of super-plasticizer and the injection pressure. Particularly, the type of super-plasticizer affected significantly the inject ability of grouts at all w/c ratios and injection pressures, as it can be seen from the initial flow rate and the passed volume, which were higher for PCE grouts than the ones obtained for SNF grouts. Moreover, it is seen that as the grout gets thicker, the effectiveness of PCE in inject ability, when compared with the one of SNF, becomes more pronounced. For example, under injection pressure of 6 bar, the passed volume of PCE grout with w/c ratio of 0.5, 0.4 and 0.33 was 41, 300 and 400% higher than the one of SNF grout, respectively. The above experimental results indicate clearly that the dispersing action of a super-plasticizer based on steric forces is more effective than the one of a super-plasticizer based on electrostatic forces, as it results in a higher initial flow rate and mainly in a more stable suspension over time, especially in the case of very dense grouts. It has long been established that when suspensions flow Brownian forces dominate and promote the particle collisions leading to coagulation and consequently to a gradual blocking of the pores of the sand by large cement particle agglomerates, which in turn obstruct the further intrusion of the suspension (Myers, 1999). The addition of surfactants that adsorb at the solid-liquid interface and provide an electrostatic or steric barrier can retard or prevent “sticky” collisions between particles, hence a more stable system. It seems that the macromolecular chains of PCE anchored to the particle

Table 4: Inject-ability characteristics of grouts

Notation	Volume of grout passed (l)	Initial flow rate (cm^3/sec)
G_1	15.1	10.00
G_2	85.0	32.50
G_3	23.3	14.00
G_4	120	50.00
G_5	0.8	2.16
G_6	12.5	24.40
G_7	2.0	3.24
G_8	50.0	34.50
G_9	Not measured	Not measured
G_{10}	8.2	22.60
G_{11}	Not measured	Not measured
G_{12}	41.0	25.00

surface form a relatively thick adsorbed layer that can impose a barrier to particle approach, giving a better protection against particle coagulation than the repulsive electrostatic forces of SNF. Additionally, an interaction between Ca^{2+} ions, stemming from pozzolanic reactions and negatively charged surface of cement particles by SNF molecules could be a reason for a reduction of repulsive electrostatic forces, resulting in the decrease of the energy barrier and facilitating particle collisions and agglomeration. This could explain why SNF appeared to be less effective in very thick grouts, since high amounts of cement increase the Ca^{2+} concentration in the system.

Figure 3 to 6 depict the change in compressive strength and elastic modulus of the grouted samples in relation to the distance from injection point. As it can be observed, the mechanical properties of injected samples decreased along the distance from the grouting

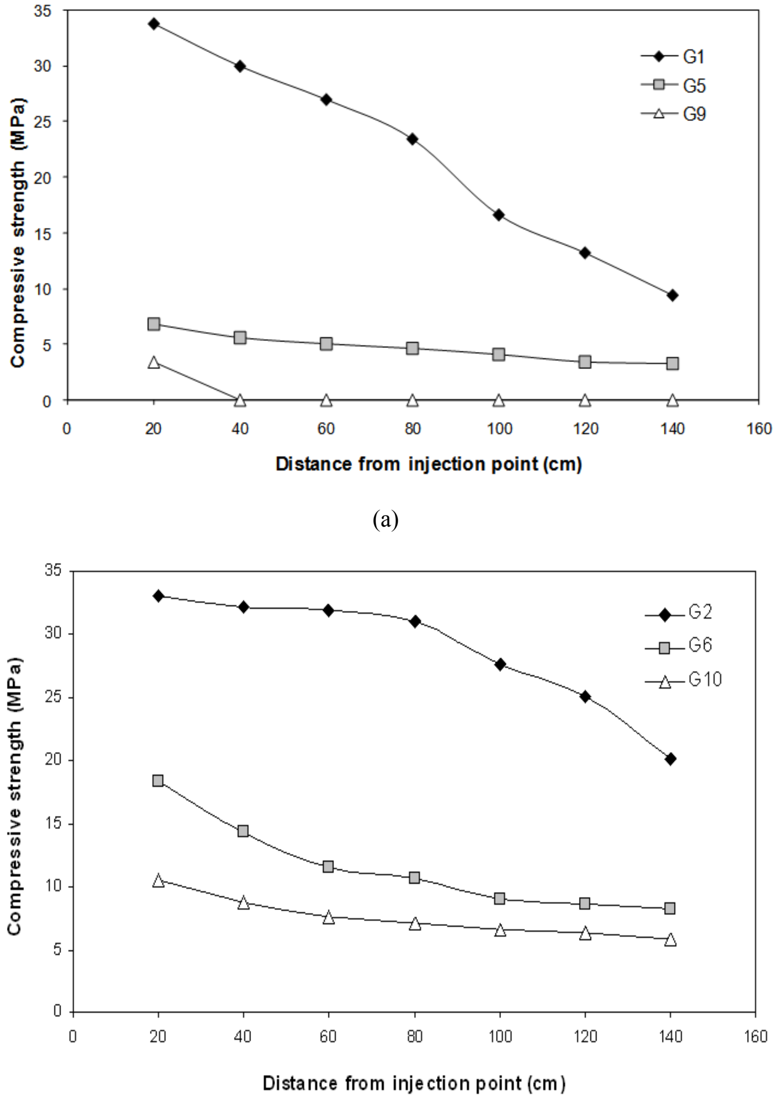

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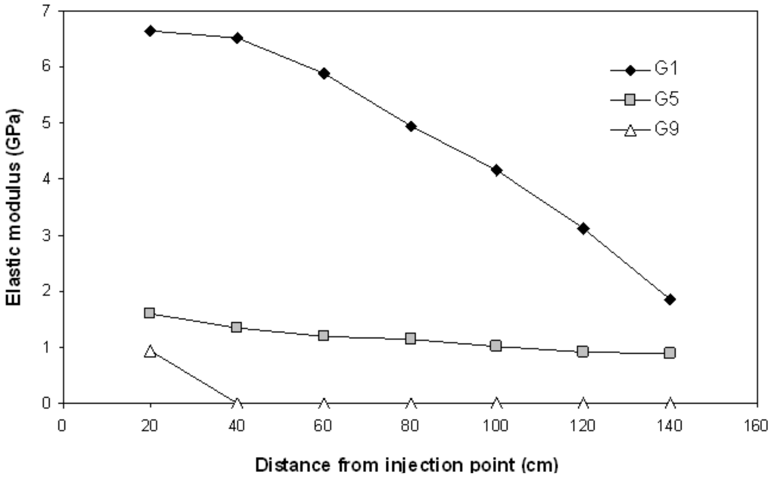
Fig. 3: Compressive strength of SNF grouted specimens plotted against distance from injection point. Injection pressure of (a) 1 bar, (b) 6 bar



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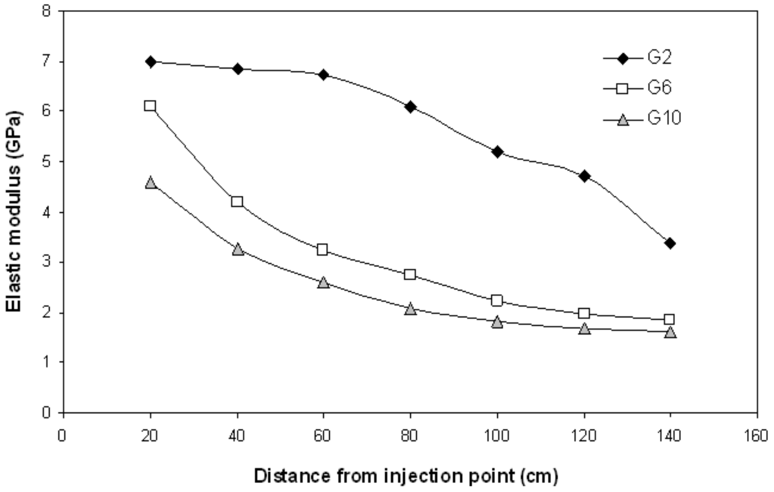

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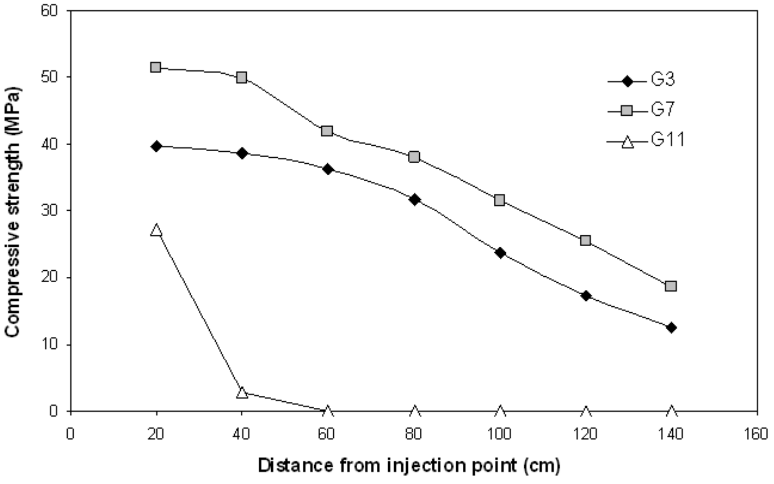
Fig. 4: Elastic modulus of SNF grouted specimens plotted against distance from injection point. Injection pressure of (a) 1 bar, (b) 6 bar



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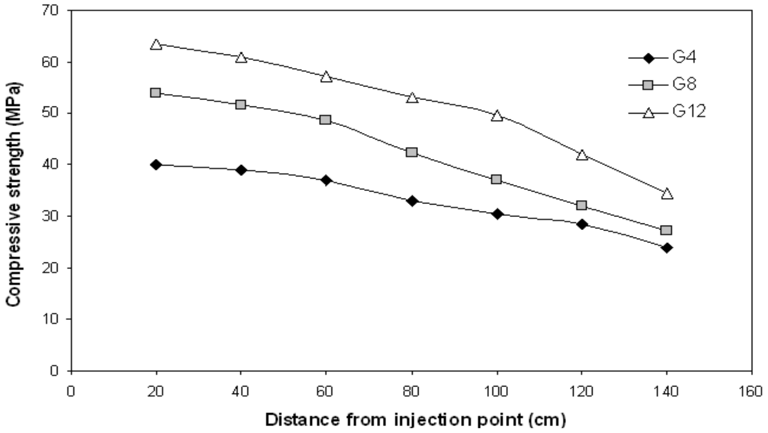

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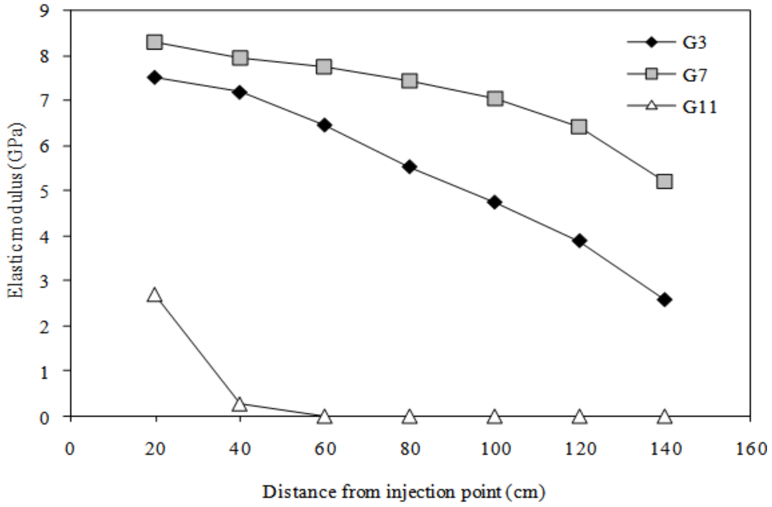
Fig. 5: Compressive strength of PCE grouted specimens plotted against distance from injection point. Injection pressure of (a) 1 bar, (b) 6 bar



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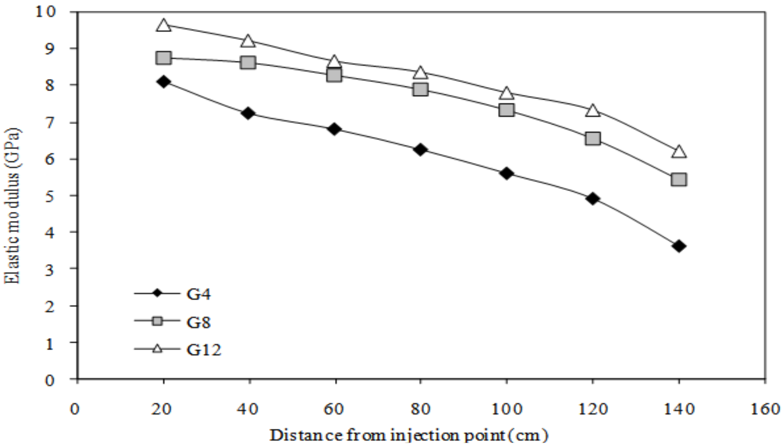
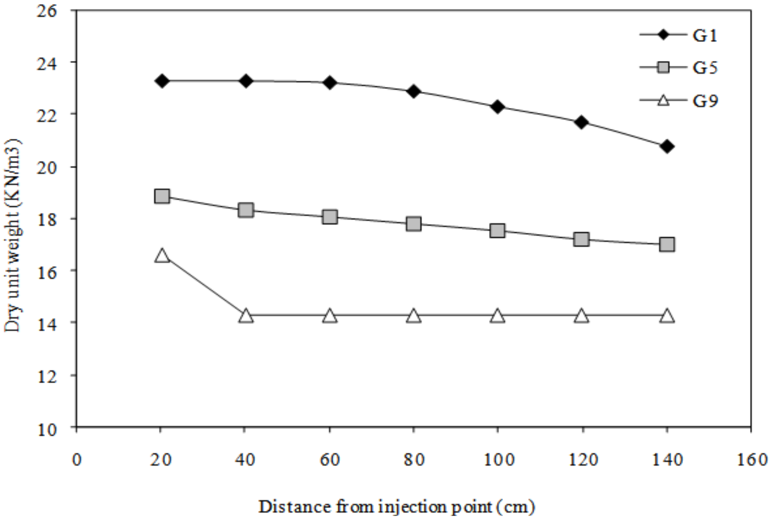


(b)

Fig. 6: Elastic modulus of PCE grouted specimens plotted against distance from injection point. Injection pressure of (a) 1 bar, (b) 6 bar

point. This is attributed to the development of filtration during the injection process, resulting in a much higher accumulation of cement particles in parts near the grouting point than in parts lain in longer distances. This tendency was more intense in the samples grouted with the low injection pressure, for both Superplasticizers. A justification for the above observation is that a low injection pressure results in a simultaneous low flow velocity which enables the action of Brownian or gravitational forces on cement particles, causing a continuous deposition of cement particles in the porous medium near the injection point, leading to the fast blocking of its voids and inhibiting the transfer of more cement amount to the rest part of the soil column. On the contrary, injection with a high pressure leads to a more effective filling of voids and to a more efficient cementation of sand grains in a longer part of soil column due to the transfer of higher amount of cement solids at higher distance from the injection point by the flowing grout during the injection process.

Unconfined compression strength tests showed that grouting with PCE grouts increased the mechanical properties of grouted samples much more than grouting with SNF grouts. For example, the compressive strength and the elastic modulus of samples injected with PCE grouts of w/c = 0.4, under injection pressure of 1 bar, were 5.7 to 8.8 times and 5.1 to 6.9 times higher than those obtained using SNF grouts, respectively. An exception is the case of grouting with grouts having w/c = 0.5, at which the strength that was achieved with SNF grouts was somewhat comparable with the one obtained with PCE grouts. The strength development of PCE and SNF grouted samples is strongly related with the w/c ratio. For PCE grouts, the



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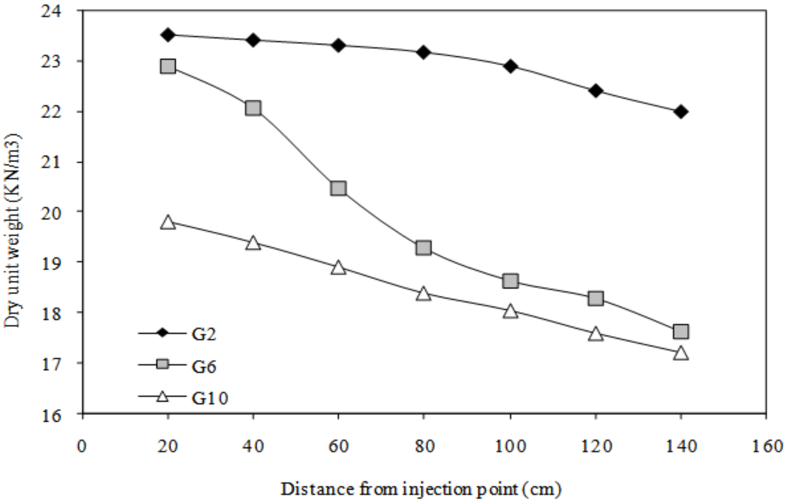

(b)

Fig. 7: Dry unit weight of SNF grouted specimens plotted against distance from injection point. Injection pressure of (a) 1 bar, (b) 6 bar

decrease of w/c ratio increased the compressive strength and elastic modulus of grouted sample, whereas for SNF grouts the decrease of w/c ratio resulted in a significant reduction of the strength parameters. This is also a strong evidence for the inability of SNF, when it is mixed with thick grouts, to disperse adequately the cement agglomerates so that to facilitate the propagation of a large amount of cement solids through porous mass. Additionally, in a considerable number of cases, it is worth noting that even in parts very close to the injection point, where the accumulated quantity of cement during the flow of the grout was expected to be high and independently of the used super-plasticizer type, the strength obtained with PCE was much higher than the one with SNF. This additional fact provides more evidence that the higher strength of PCE grouted samples depends on the aforementioned dispersion mechanism and the consequential better hydration of cement particles by the action of PCE macromolecular chains.

The porosity n and dry unit weight γ_d of grouted soil samples are depicted in Fig. 7 to 10. As for the mechanical properties, the values of γ_d and n were also dependent directly on injection pressure, w/c ratio and super-plasticizer type. A large difference was observed in γ_d and n of the samples grouted with PCE grouts of w/c = 0.4 and 0.33 with those of samples grouted with SNF grouts, whereas in the case of grouting with grouts of w/c = 0.5 this difference was negligible. The comparison of the above experimental results confirms that as the grout becomes thicker, SNF becomes ineffective in transferring large quantities of cement through soil mass, leading to a partial filling of voids (low values of γ_d and high values of n) and to a loose

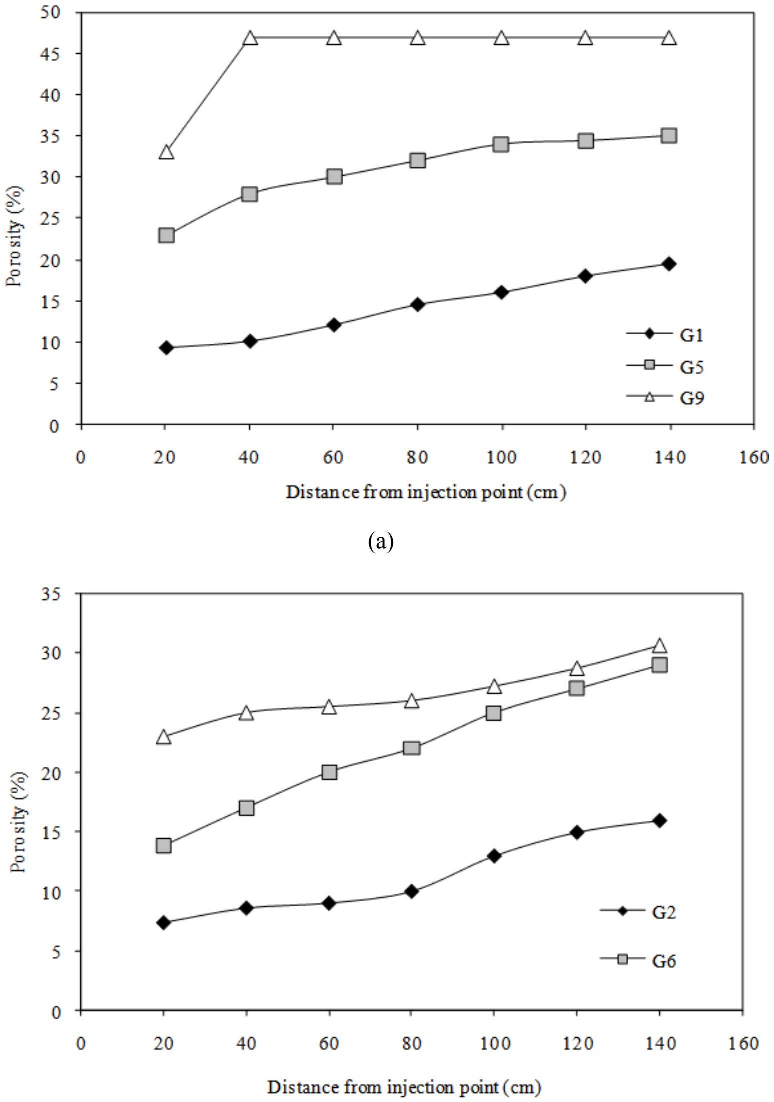

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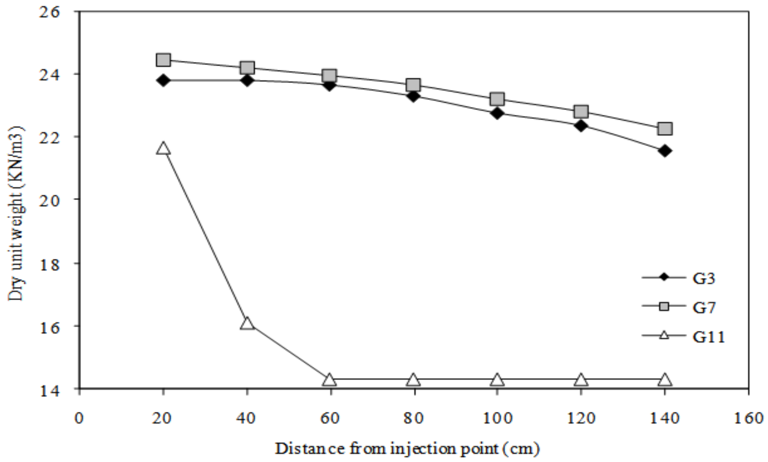
Fig. 8: Porosity of SNF grouted specimens plotted against distance from injection point. Injection pressure of; (a): 1 bar; (b): 6 bar



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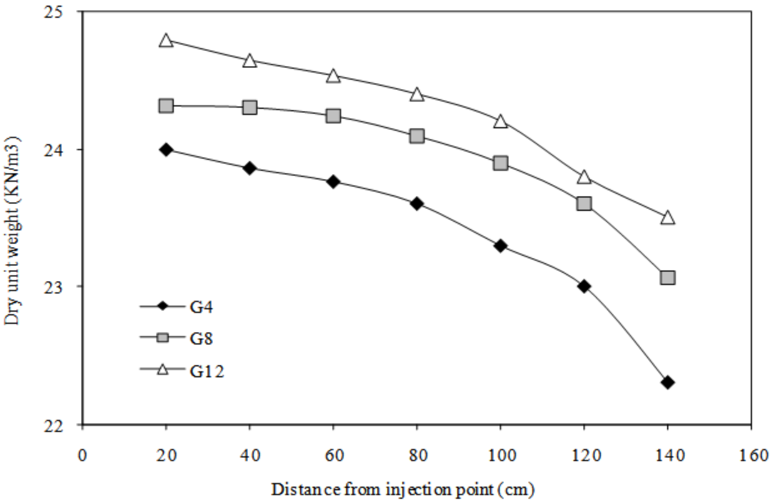

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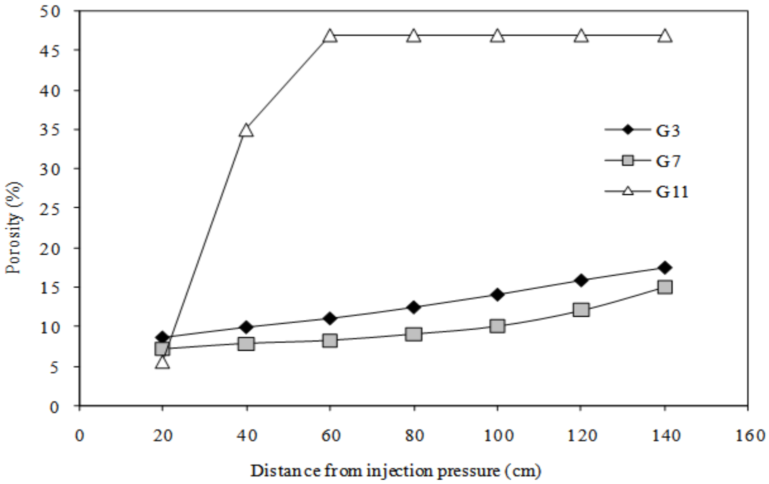
Fig. 9: Dry unit weight of PCE grouted specimens plotted against distance from injection point. Injection pressure of, (a) 1 bar, (b) 6 bar



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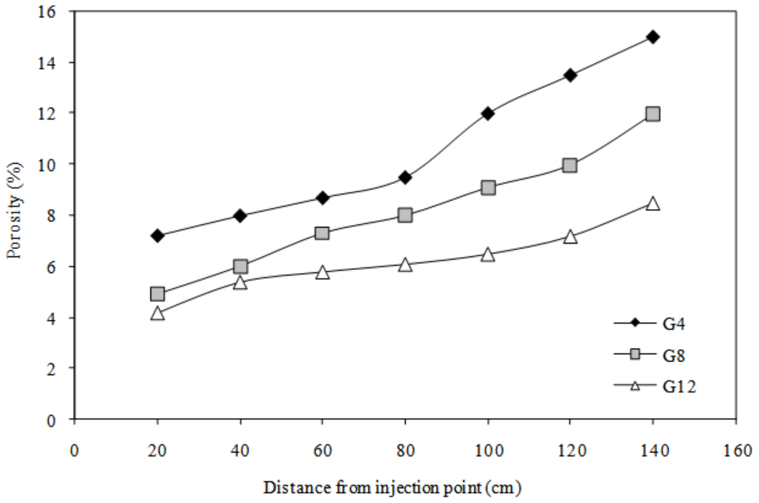

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Fig. 10: Porosity of PCE grouted specimens plotted against distance from injection point. Injection pressure of, (a) 1 bar, (b) 6 bar

Table 5: Permeability test results and values of permeability coefficient k (m/sec) of grouted soil specimens in relation to the distance from injection point

	Distance from injection point (cm)						
Notation	0-20	20-40	40-60	60-80	80-100	100-120	120-140
G_1	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_2	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_3	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_4	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_5	8.8×10^{-4}	1.4×10^{-3}	1.6×10^{-3}	1.8×10^{-3}	2×10^{-3}	2.2×10^{-3}	2.3×10^{-3}
G_6	5.3×10^{-4}	6.3×10^{-4}	6.4×10^{-4}	6.5×10^{-4}	7.8×10^{-4}	1×10^{-3}	1.8×10^{-3}
G_7	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_8	No flow	No flow	No flow	No flow	No flow	No flow	No flow
G_9	6.5×10^{-4}	4×10^{-2}	4×10^{-2}	4×10^{-2}	4×10^{-2}	4×10^{-2}	4×10^{-2}
G_{10}	4.5×10^{-4}	5.2×10^{-4}	7.3×10^{-4}	1.6×10^{-3}	1.8×10^{-3}	2.1×10^{-3}	3×10^{-3}
G_{11}	No flow	3×10^{-3}	4×10^{-2}	4×10^{-2}	4×10^{-2}	4×10^{-2}	4×10^{-2}
G_{12}	No flow	No flow	No flow	No flow	No flow	No flow	No flow

structure between soil-cement particles with a subsequent low strength. On the other hand, the higher dispersive action of PCE induces the transferring of more cement particles into the soil mass, improving the packing of the soil matrix and forming a denser composite structure (high values of γ_d and low values of n) with higher strength. The similar values of γ_d and n obtained from samples grouted with PCE or SNF grouts with w/c = 0.5, examined in respect to the strength test results, imply the better hydration of cement that occurs in the presence of PCE molecules, which results in an improved strength.

The results of permeability tests are summarized in Table 5, where permeability coefficient k is presented as a function of the distance from injection point. No flow of water was observed through grouted specimens with PCE grouts, a fact that indicates the significant improvement of the pore structure of the granular soil achieved by the use of this kind of super-plasticizer at any w/c ratio. On the contrary, permeability of grouted samples with SNF grouts was strongly related with w/c ratio and injection pressure. k exhibited a trend to increase in all grouted samples as w/c ratio and injection pressure decreased. An exception is the case of grouts with w/c = 0.5 at which the grouted specimens appeared to be also impermeable.

CONCLUSION

In this comprehensive laboratory study, the effect of super-plasticizer type on grouting with thick cement suspensions was investigated. In particular, the following conclusions can be drawn:

- The inject ability of grouts super-plasticized with PCE, a new generation of polycarboxylate super-plasticizer acting by steric effect, is higher than the one of grouts mixed with SNF. The efficacy of PCE on inject ability of grouts is more pronounced when w/c ratio decreases.

- Grouted samples with PCE grouts appear to have an increased compressive strength and elastic modulus when compared with samples treated with SNF grouts. For PCE grouts, the decrease of w/c ratio results in an appreciable increase of compressive strength and elastic modulus, whereas in the case of SNF grouts the decrease of w/c ratio causes a significant reduction of strength parameters.
- The experimental results obtained for the physical parameters of grouted samples confirm that due to the higher dispersive action of PCE more cement quantity is transferring into the soil mass, resulting in a more efficient grouting.

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