Cement & Concrete Composites 34 (2012) 593–603

Contents lists available at SciVerse ScienceDirect

Cement & Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Development of microfine cement grouts by pulverizing ordinary cements

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ARTICLE INFO

Article history:
Received 23 December 2010
Received in revised form 24 January 2012
Accepted 25 January 2012
Available online 14 February 2012

Keywords:
Grouting
Microfine cements
Pulverization
Suspension properties
Groutability
Effectiveness

ABSTRACT

Three different cements (CEM I, CEM II/B-M and CEM IV/B according to EN 197-1) containing 0%, 23.5% and 38% of pozzolan, respectively, were pulverized to obtain three additional gradations from each cement, with nominal maximum grain sizes of 40, 20 and 10 μm. Cements with the two finer gradations are classified as ''microfine'' cements. Suspension properties, groutability and effectiveness of all cements were evaluated for water-to-cement ratios (W/C) of 1, 2 and 3 by weight. A superplasticizer was used to optimize rheological properties. The properties and performance of all suspensions tested are affected primarily by W/C ratio and cement fineness. All microfine cement suspensions have acceptable apparent viscosity, behave as Bingham fluids, are stable for W/C = 1, have reasonable setting times for field applications, have mostly predictable groutability and provide satisfactory strength to grouted sands. The finer gradations of II/B-M cement exhibited the best overall behavior and are considered as the most promising compared to similar gradations of the other two cements.

1. Introduction

Improvement of the mechanical properties and behavior of soils by permeation grouting, using either suspensions or chemical solutions, is frequently required in order to assure the safe construction and operation of many structures. Suspensions have lower cost and are harmless to the environment but cannot be injected into soils with gradations finer than coarse sands. Chemical solutions can be injected in fine sands or coarse silts but are more expensive and, some of them pose a health and environmental hazard. Efforts have been made to extend the injectability range of suspension grouts by developing materials with very fine gradations. As a result, a number of ''microfine'' and ''ultrafine'' cements has been developed and marketed. Although the two terms imply gradation differences, no generally accepted definitions have been established to date and the terms are used interchangeably. According to EN 12715, ''microfine'' cements should have $d_{95} < 20$ μm and a Blaine fineness value over 800 m^2/kg. The International Society for Rock Mechanics, the American Concrete Institute Committee 552 and the Portland Cement Association define ''microfine'' cements as having, respectively, $d_{95} < 16$ μm, $d_{max} < 15$ μm and $d_{max} < 10$ μm. Some National Standards define ''microfine'' as having $d_{95} < 30$ μm and ''ultrafine'' as having $d_{95} < 15$ μm or even $d_{max} < 6$ μm [1–4]. The term ''microfine'' will be used in this paper.

Available injectability criteria based on soil and grout characteristic grain sizes [5] indicate that a reduction of the larger grain sizes of the grout (d_{85} or d_{95}) by one order of magnitude may result in a similar reduction of the characteristic grain sizes (d_{15} or d_{10}) of groutable soils. Other than this obvious advantage, use of microfine cements compared to ordinary, coarse-grained, cements: (a) has detrimental effects on viscosity and rheological properties which may be negated by increasing the water-to-cement ratio, W/C, or by using an appropriate additive, such as a superplasticizer, (b) has a positive effect on suspension stability (bleed capacity and bleeding rate) yielding stable suspensions with up to double the W/C ratio, (c) results in decreased setting times which, when prohibitively low, may be corrected by using a superplasticizer or increasing the W/C ratio, (d) has an effect on grout strength which increases for W/C > 1 but may decrease for W/C < 1, (e) yields higher unconfined compression strength of grouted soils and (f) yields higher permeability reductions [1,6–12].

The experimental investigation reported herein is part of an extensive research effort aimed toward the development of a relatively fine-grained material, suitable for suspension-type permeation grouting and obtained by pulverization of ordinary cements produced in Greece. Accordingly, suspensions of three different cement types, each at four different gradations, were tested. The results obtained allow comparisons to be made between the behavior and performance of fine-grained versus coarse-grained cement grouts and an evaluation of the cement type and fineness that is most promising for development and marketing as a

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doi:10.1016/j.cemconcomp.2012.01.009

microfine cement. According to standard practice, the documentation and optimization of suspension properties are based on measured rheological properties, bleeding behavior, setting times and strength development. To provide full documentation, information on the injectability and effectiveness of these suspension grouts is presented.

2. Materials

For the purposes of this investigation, three cement types (Portland, Portland-composite and pozzolanic cement, code-named CEM I, CEM II/B-M and CEM IV/B, respectively, according to EN 197-1) were selected because of production cost differences. The compositions of these cements are presented in Table 1 both in terms of oxides and in terms of the raw materials used for their production. The amount of clinker used for the production of the CEM I cement is significantly higher in comparison to the other cements which are more pozzolanic. The chemical analysis of the cements reflects the anticipated differences in oxide composition according to cement type. Each ordinary cement was pulverized to produce three additional cements with nominal maximum grain sizes (d_{max}) of 40 μm, 20 μm and 10 μm. A special laboratory mill, based on dry grinding with colliding air jets and equipped with a high-speed separator, was used to pulverize the cements.

The grain size distributions of these cements were obtained using the laser diffraction method, according to ISO 13320-1, and typical gradations are shown in Fig. 1. Characteristic grain sizes (ISO 13320-1) and Blaine specific surface values (EN 196-6) for all cements are presented in Table 2. In terms of gradation, all cements with nominal d_{max} = 10 μm can be considered as "microfine" since they satisfy the requirements of Standard EN 12715 (d_{95} < 20 μm and specific surface over 800 m^2/kg) as well as definitions adopted by various organizations. Also, cements with nominal d_{max} = 20 μm have adequately small characteristic grain sizes to be considered, marginally, as "microfine". Presented in Fig. 2 is the envelope of the gradations of eighteen fine-grained cements, as reported in available literature or in the official sites of the producers, together with the envelope of the gradations of the six finer cements, with nominal d_{max} of 20 μm and 10 μm, which were used in this investigation. It can be observed that the gradations of the fine-grained cements produced by pulverization are within the range defined by a large number of available fine-grained cement products.

All suspensions tested during this investigation were prepared using potable water since it is considered appropriate for preparing cement-based suspension grouts [13,14]. The water-to-cement ratio (W/C) of the suspensions was set equal to 1, 2 and 3 by weight, because suspensions with a W/C > 3 would have prohibitively large bleeding, long setting times and low strengths, while suspensions with a W/C < 1 would have prohibitively high viscosity [13,15,16]. A superplasticizer was used to improve the suspension properties of the pulverized cements. This superplasticizer is a patented new generation of admixture based on polycarboxylate chemistry.

Table 1
Composition of cements.

Oxides (%)	Cement type		
Components (%)	I	II/B-M	IV/B
SiO_2	19.38	26.67	29.99
Al_2O_3	4.28	7.46	11.22
Fe_2O_3	3.24	4.30	3.83
CaO	64.11	49.37	47.92
MgO	3.43	3.54	2.14
K_2O	0.57	0.77	0.95
Na_2O	0.17	0.34	0.38
Na_2Oeq	0.55	0.85	1.10
SO_3	3.09	3.07	2.60
LOI	3.73	6.06	2.56
Clinker	90.3	63	58
Limestone	4.7	8.5	2
Pozzolan	0	13.5	20
Fly ash	0	10	18
Gypsum	5	5	2

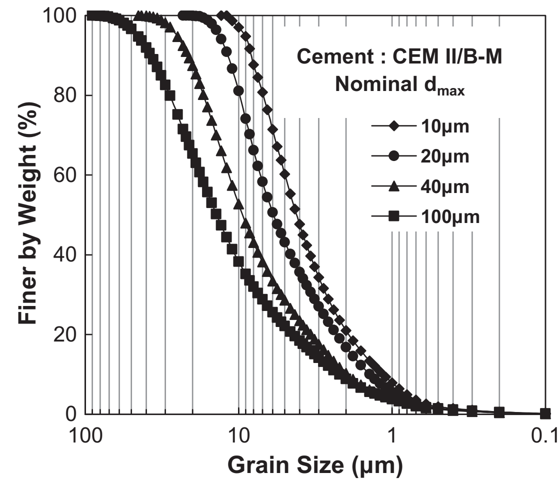

Fig. 1. Typical cement gradations.

Table 2
Gradation of cements.

Cement type	Grain sizes (μm)				Blaine (m^2/kg)
	d_{max}[a]	d_{95}	d_{50}	d_{10}	
I	100	57.0	16.6	3.0	384
	40	22.5	8.6	2.0	529
	20	11.5	4.2	1.2	710
	10	8.2	3.2	1.0	920
II/B-M	100	45.5	14.0	2.2	466
	40	25.8	9.4	2.0	591
	20	13.6	5.8	1.4	735
	10	9.1	4.2	1.1	942
IV/B	100	48.0	14.2	3.0	452
	40	26.0	9.3	2.2	582
	20	12.8	4.4	1.3	715
	10	9.8	3.9	1.2	923

[a] Nominal maximum cement grain size.

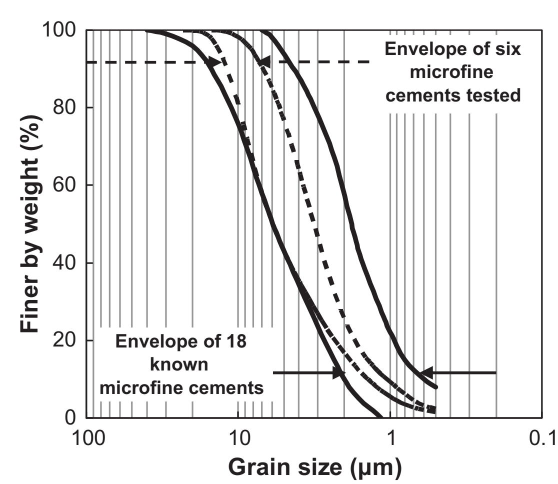

Fig. 2. Comparison of cement gradations.

The soils used for injectability evaluation were clean, uniform, limestone sands with angular grains. Five different sand gradations were used with grain sizes limited between sieve sizes (ASTM E11) Nos. 5 and 10, 10 and 14, 14 and 25, 25 and 50, and 50 and 100 having, respectively, a d_{15} size of 2.2 mm, 1.5 mm, 0.8 mm, 0.34 mm and 0.17 mm. Effectiveness evaluation was based on the unconfined compression strength of some of these sands grouted, in a dense and dry state, with various grout suspensions. The void ratio of the clean sands ranged between 0.67 and 0.70 corresponding to a relative density value of approximately 90–95%. All suspensions used for injectability and effectiveness evaluations contained superplasticizer.

3. Experimental procedures

All suspensions were prepared using two high speed mixers, of the type used for the preparation of soil specimens for hydrometer testing, with a speed of 10000 rpm at no load. The water for suspension preparation was stored at room temperature for at least 48 h before use. Accordingly, the temperature of the water was practically constant and equal to 23 ± 2 °C. For suspensions with superplasticizer, the appropriate amount of cement and 70% of the required water were placed in the mixer together with the superplasticizer dosage and mixed for 5 min. Then, the rest of the water was added and mixing continued for another 5 min. This procedure was recommended by the superplasticizer producer. For reasons of uniformity, the same total mixing time of 10 min was also used for the preparation of suspensions without superplasticizer, after placing the appropriate amounts of water and cement in the mixer. Mixing resulted in reasonably increased suspension temperatures of 28 ± 4 °C, immediately after preparation. Suspension temperatures gradually decreased to average values of 26.5 °C (range from 22.5 °C to 29 °C), 25.5 °C (range from 21.5 °C to 27.5 °C) and 24.5 °C (range from 20.5 °C to 27 °C) at elapsed times of 30 min, 60 min and 120 min from suspension preparation, respectively. All the aforementioned temperatures were not affected by the use of superplasticizer and are considered as not excessive.

Viscosity and rheological properties of the suspensions were investigated using a rotational viscometer equipped with coaxial cylinders for viscosity measurements between 1 and 10 mPa s and spindles (cylindrical or disc-shaped) for measurements between 10 and 10^6 mPa s. The instrument displays a viscosity value computed by assuming that the fluid is Newtonian. When the tested fluids are non-Newtonian, the instrument displays an "apparent viscosity" reading. Immediately after preparation, the suspension was placed in a 1 l container with a diameter of 104 mm and the initial viscosity value was measured (time from preparation equal to 0 min). Subsequent measurements were taken at predetermined time intervals, up to 180 min from preparation, at spindle or coaxial cylinder rotation speeds equal to 60, 30, 12, 6 and 3 rpm, in order to obtain information on the rheological properties of the suspensions. Between viscosity readings, the suspensions were continuously agitated with a low speed mixer to retain homogeneity and avoid bleeding. Rheological curves (flow curves), representing the variation of shear stress as a function of shear strain rate, were computed according to the methodology proposed by Krieger and Maron [17] since it is considered to yield limited error in comparison to other available methodologies.

Bleeding rate and bleed capacity were investigated by conducting sedimentation tests. Immediately after suspension preparation, 1 l of the suspension was placed in a graduated cylinder with a diameter of 63 mm and the volume of sediment was recorded as a function of time for a period of up to 24 h to ensure complete sedimentation. Bleeding, at any time after preparation, is defined as the volume of bleed water above the suspension, ΔV, expressed as a percentage of the total initial volume of the suspension, V_0. The final value of $\Delta V/V_0$ (after complete sedimentation) is defined as the bleed capacity of the suspension.

Setting times and early strength development were investigated by conducting Vicat needle tests and pocket penetrometer tests, respectively. According to ASTM Standard C191, initial setting time is the time from suspension preparation when the penetration of the Vicat needle in the specimen is equal to 25 mm and final setting time is the time when the penetration of the Vicat needle in the specimen is negligible (considered to be equal to 1 mm for the purposes of this investigation). Specimens for Vicat needle tests were formed by placing the suspension in the standard cell on which an extension had been attached. The initial volume of the suspension was larger than the cell volume to allow for bleeding. After completion of bleeding, the bleed water, the cell extension and the excess sediment were removed, leaving the cell full with sediment to be tested.

Early strength was measured using a pocket penetrometer, which yields a direct estimation of unconfined compression strength up to a maximum value of 450 kPa. Specimens for strength testing were prepared by placing the suspension in cylindrical containers. After completion of bleeding, the bleed water was removed carefully and measurements were started. Between measurements, the upper surface of all specimens was sealed with a plastic membrane to avoid water loss by evaporation.

Strength development was investigated by conducting unconfined compression tests at elapsed times of 1, 3, 7, 14 and 28 days from suspension preparation. Specimens ready for unconfined compression testing were prepared by placing the suspension in split, cylindrical, Teflon molds, with a diameter equal to 50 mm and a height of 110 mm, equipped with an extension to allow for bleeding. After 24 h, the specimens were extracted from the split molds, placed in plastic bags to avoid significant variations of moisture content and cured in a humid room at 20 ± 3 °C and relative humidity above 95% for the predetermined time. For the elapsed time of 1 day, specimens were tested immediately after extraction from the split molds. For cases with final setting times over 24 h, testing for short elapsed times was not possible. An automatic loading machine with maximum capacity of 100 kN was used for testing at an axial strain rate equal to 0.05%/min.

Injectability of the suspensions was evaluated by performing injections into columns of dense, dry sand with a diameter of 75 mm and a height of approximately 400 mm. A special assembly, consisting of a pressurized feed tank with a stirring shaft, an air pressure regulator and a line to the PVC grouting column, was used. Injection was stopped when either the volume of the injected grout was equal to two void volumes of the sand in the column or when the injection pressure became equal to 200 kPa.

Effectiveness of the suspensions was evaluated by performing unconfined compression tests on grouted sands. The sands were at a dense and dry state before grouting. Laboratory equipment, similar to the arrangement described in ASTM D4320-84, was used to produce small-size grouted sand specimens, with a height of 112 mm and a diameter of 50 mm, ready for testing. Injection was stopped when either the volume of the injected grout was equal to two void volumes of the sand in the mold or when the injection pressure became equal to 200 kPa. After curing for 28 days, the grouted sand specimens were tested at an axial strain rate equal to 0.05%/min.

4. Apparent viscosity

Apparent viscosity values were initially obtained for all suspensions which were prepared without using any additives. Typical results of apparent viscosity values as a function of time, for a rotation speed of 60 rpm, are presented in Fig. 3a. One hour after suspension preparation, the increase of the apparent viscosity value ranged between 15% and 65% with an average of 40%. At 3 h after suspension preparation, the increase of the apparent viscosity value ranged between 30% and 130% with an average of 74%. Presented in Fig. 3b are the apparent viscosity values of all suspensions as measured immediately after preparation (elapsed time 0 min, rotation speed of 60 rpm). It can be observed that apparent viscosity values decrease, on the average, by one order of magnitude as the W/C ratio increases from 1 to 3, regardless of cement type or fineness. In general, the cement type has an effect on the apparent viscosity values, with CEM IV/B cement suspensions yielding higher values and CEM II/B-M cement suspensions yielding lower values. Concentrating on the fine-grained suspensions (nominal d_{max} of 20 μm and 10 μm) immediately after preparation, the apparent viscosity values of the CEM II/B-M suspensions are, on the average, lower than CEM I and CEM IV/B values by 40% and 52%, respectively.

In terms of cement gradation, it can be observed that the first stage of cement pulverization, from nominal d_{max} = 100 μm to 40 μm, results, generally, in a small increase of the apparent viscosity of the suspensions by 10–50% with an average of 25%. However, cement pulverization to a nominal d_{max} = 20 μm or 10 μm, results in a significant increase of the apparent viscosity of the suspensions by a factor of 3–9 with an average of 5. As frequently reported in available literature i.e. [7,8,18], the use of fine-grained cements has a strong effect (significant increase) on suspension apparent viscosity compared to similar, in terms of W/C ratio, ordinary cement suspensions. This effect is attributed to the increase of the surface area of the reaction media and, consequently, to faster reaction (faster hydration) and "thickening" of the suspension.

It is generally desirable that cement suspension grouts have reasonably low viscosity in order to enhance pumpability and groutability. Although limiting values have not been established, it is frequently recommended that initial viscosity values should range between 30 mPa s and 50 mPa s [19]. A review of the apparent viscosity values obtained for the suspensions, and especially those for W/C = 1, indicates that they are prohibitively high for permeation grouting regardless of cement type or fineness. Accordingly, it was decided to use a superplasticizer for improvement. The effect of the superplasticizer was quantified for four different dosages equal to 0.6%, 1.0%, 1.4% and 1.8% by weight of dry cement, since the producer of the superplasticizer recommends a dosage between 0.53% and 1.91%. The four superplasticizer dosages were tested on suspensions of all three cement types at the finer nominal d_{max} of 10 μm and 20 μm and at W/C = 1. Typical results are presented in Fig. 4. It can be observed that the apparent viscosity values decrease with

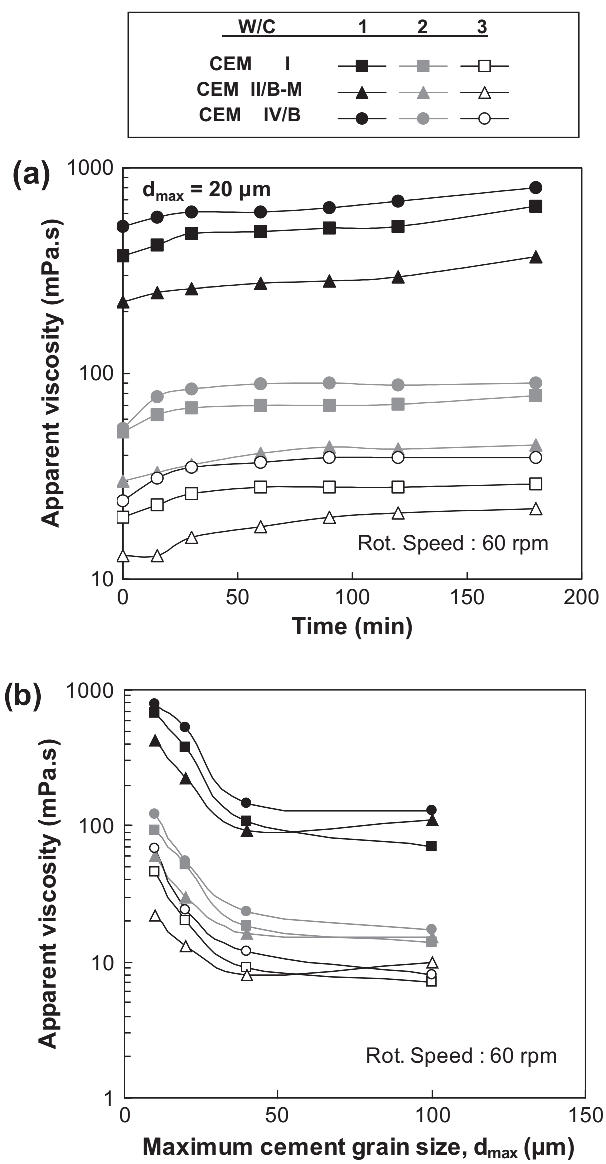

Fig. 3. Apparent viscosity values of cement suspensions without additive (a) typical variation with time and (b) values at 0 min elapsed time.

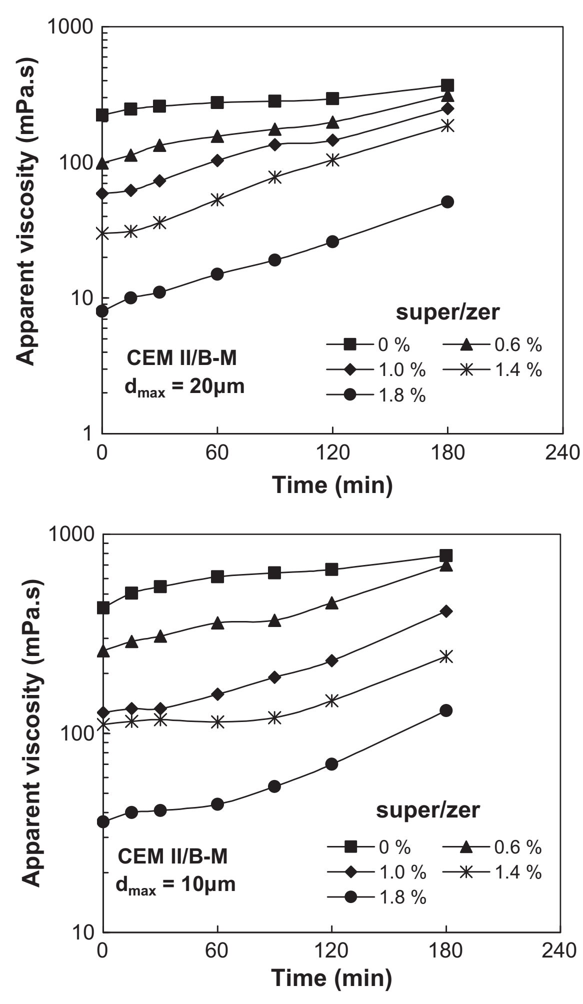

Fig. 4. Effect of superplasticizer dosage on apparent viscosity.

increasing superplasticizer content, regardless of cement fineness. For all suspensions tested at elapsed times of 0 min and 15 min from preparation and at viscometer rotation speeds equal to 60, 30 and 12 rpm (30 viscosity measurements), the average decrease of the apparent viscosity values was 44%, 69%, 80% and 93% with standard deviation of 15%, 11%, 10% and 5%, for superplasticizer dosage of 0.6%, 1.0%, 1.4% and 1.8%, respectively. Based on the desire to achieve low initial apparent viscosity values and to reduce negative effects on other suspension properties if the maximum recommended dosage was applied, it was decided to use a superplasticizer dosage of 1.4% by weight of dry cement and to apply the same dosage to all cement suspensions for reasons of uniformity and preparation simplicity, without optimizing the dosage for each specific suspension. The rationale of uniform superplasticizer dosage regardless of suspension W/C ratio was also adopted by other researchers [20–22].

Using the fixed superplasticizer dosage, measurements of the apparent viscosity and determination of the rheological properties of all cement suspensions were made. As indicated in Fig. 5a, the apparent viscosity of cement suspensions with superplasticizer has, qualitatively, the same behavior as the apparent viscosity of cement suspensions without superplasticizer, increasing with time and with decreasing W/C ratio. More specifically, the decrease of W/C ratio from 3 to 2 and 1 leads to a maximum increase of apparent viscosity values by 7.5 times and 56 times and to an average increase by 1.5 times and 17 times, respectively, regardless of cement type and fineness. The effect of cement type is inconsistent. Pulverization of the cements to nominal d_{max} = 20 μm and 10 μm has a strong effect on suspension apparent viscosity since it results in a maximum increase of viscosity values by 5.5 times and 48 times with an average of 1.5 and 6 times, respectively, regardless of cement type and suspension W/C ratio. Comparing suspensions of ordinary and fine-grained cements (d_{max} = 100 μm and d_{max} = 10 μm) for increasing W/C ratios 1, 2 and 3, the average increase of the apparent viscosity is equal to 14, 2.5 and 1.5 times (maximum increases of 48, 4.5 and 3 times), respectively. This indicates the significant but opposite effect of W/C ratio and pulverization which may negate each other. As shown in Fig. 5b, the apparent viscosity of all suspensions with the fixed superplasticizer dosage, as measured immediately after preparation for a rotation speed of 60 rpm, has low values ranging below 10 mPa s for suspensions with W/C = 2 and 3 and below 100 mPa s (CEM II/B-M) or 200 mPa·s (CEM I and CEM IV/B) for suspensions with W/C = 1.

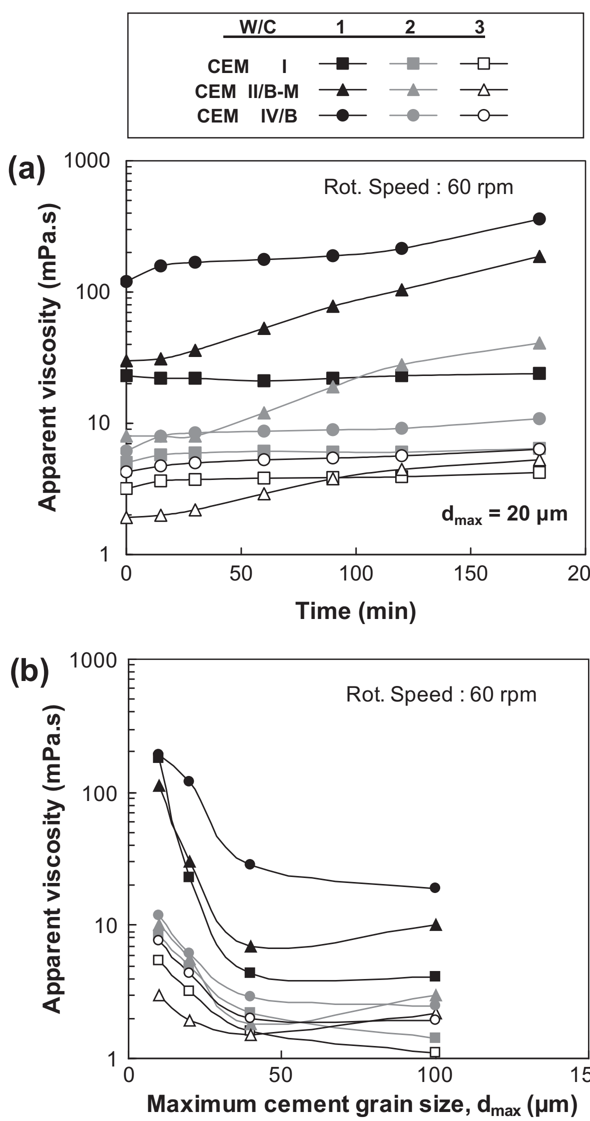

Fig. 5. Apparent viscosity values of cement suspensions with superplasticizer (a) typical variation with time and (b) values at 0 min elapsed time.

5. Rheological properties

In terms of rheological behavior, all suspensions tested behaved, generally, as non-Newtonian fluids, since the apparent viscosity values were dependent on the rate of shear strain. Typical

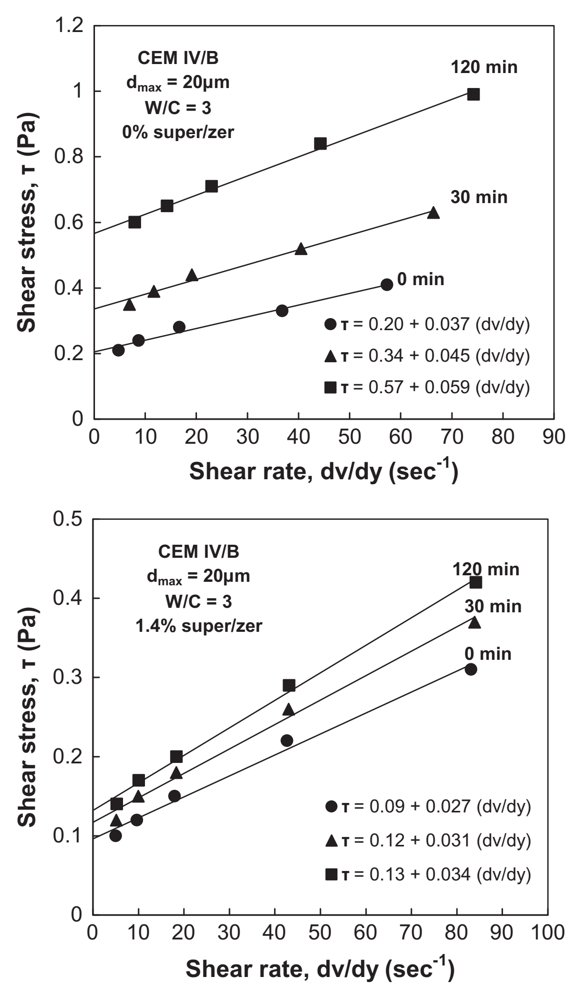

Fig. 6. Typical flow curves of cement suspensions with and without superplasticizer.

rheological curves (down curves) of cement suspensions with or without superplasticizer are presented in Fig. 6. The rheological curves of all suspensions, tested with or without superplasticizer, can be described either as straight lines intersecting the shear stress axis or as having an initial, short, curved section followed by a straight line. Ideal Bingham fluids present a clearly defined initial yield stress and linear rheological curves. However, real fluids may not exhibit such well defined yield stresses and, sometimes, may have initially curved rheological curves. This behavior was observed in a limited number of cases, for coarse-grained cement (d_{max} = 100 μm and 40 μm) suspensions without superplasticizer, and can be attributed either to the true behavior of fluids [23] or to the principle of operation of the coaxial cylinder viscometers [24]. Therefore, the fitting of the experimental data with a linear rheological curve was applied, in these few cases, without considering the measurement corresponding to the minimum shear rate and, in all the other cases, using all measurements. As a result, the values of the Bingham model parameters as well as the correlation coefficients, R^2, of the Bingham model to the experimental data, for all suspensions prepared with or without superplasticizer, immediately after preparation, are presented in Table 3. For suspensions with high apparent viscosity, where a disc-shaped spindle was used, no attempt was made to obtain a flow curve because the shear rate is hard to define since it varies across the disc [25] and, consequently, the relevant parameter values are not included in Table 3. The values of correlation coefficient range from 0.95 to 0.99 and are in many cases, especially for suspensions with superplasticizer, equal to 0.99 indicating a satisfactory fitting of the Bingham model to the experimental data. In grouting practice, cement suspensions are usually described as Bingham fluids i.e. [7,10,13,16,18,26]. Although the Bingham model is simplified, it is considered as adequately effective and particularly practical in grouting applications [7]. Taking into consideration all these factors, it can be stated with confidence that the cement suspensions tested during this investigation behaved as Bingham fluids.

As typically shown in Fig. 6, the yield stress (intersection of shear stress axis with the flow curve), τ_o, obtained by performing dynamic measurements [27], increases with time while the plastic viscosity (slope of the flow curve), n_p, remains practically constant. The W/C ratio affects the values of the Bingham model rheological parameters, since an increasing W/C ratio results, generally, in decreased yield stress and plastic viscosity (Table 3). Furthermore, it is evident that use of the superplasticizer reduces the values of the Bingham model parameters. For example, the yield stress of suspensions without superplasticizer with d_{max} = 20 μm and 10 μm at W/C = 2 ranged between 0.28 and 0.60 Pa and between 0.70 and 1.32 Pa while, for similar suspensions with superplasticizer, the range was 0.04 to 0.20 Pa and 0.20 to 0.48 Pa, respectively. In terms of plastic viscosity, the respective ranges were 4.1 to 5.4 mPa s and 3.2 to 4.6 mPa s without superplasticizer and 2.7 to 3.3 mPa s and 1.1 to 3.9 mPa·s with superplasticizer. The effect of superplasticizer is more pronounced on yield stress values rather than on plastic viscosity values. The yield stress of coarse-grained suspensions (d_{max} of 100 μm and 40 μm) is reduced by even more than one order of magnitude while that of the fine-grained suspensions (d_{max} of 20 μm and 10 μm) was reduced by a factor of 3–7. In contrast, the reduction factor of plastic viscosity ranged between 1 and 5 with an average of 2.4. The predominant effect of superplasticizers on the yield stress is also reported in the available literature for cement pastes [28] and for fresh concretes [29] and can possibly be attributed [24] to the interaction of superplasticizers with the cement particles. An adsorbed skin is introduced that prevents close contact between the particles due to a combination of electrostatic and steric repulsion, weakening the "structure" which can form at rest and reducing the yield stress. Since the particles are dispersed there is a small change in plastic viscosity but this depends on the overall particle size distribution of the concrete mix. It should be noted that use of superplasticizer, for some suspensions and for a short time from preparation, resulted in very small values for the yield stress allowing an assumption of Newtonian fluid behavior for modeling. Finally, it should be noted that the CEM II/B-M cement suspensions, which generally exhibited the best behavior in terms of apparent viscosity, had generally the lowest values of yield stress and plastic viscosity compared to CEM I and CEM IV/B cement suspensions, especially for suspensions with d_{max} = 20 μm and 10 μm.

6. Bleeding

According to European Standard EN 12715, a suspension is characterized as "stable" if it has a bleed capacity of up to 5% after 120 min from preparation. It is considered good practice to use stable suspensions because unstable suspensions may provide only a partial filling of soil voids due to bleeding [9]. In general, grouts based on ordinary cements are stable when the W/C ratio is less than 0.85, while fine-cement based grouts are stable for W/C ratios up to 1.6 [7,8]. Summarized in Table 4 are the bleed capacity values of all suspensions tested, prepared with or without superplasticizer. Bleed capacity increased with increasing W/C ratio, as reported in available literature [30,31]. Stable suspensions, having bleed

Table 3
Rheological properties of cement suspensions.

d_{max} (μm)	W/C	Without additives									1.4% Superplasticizer								
		CEM I			CEM II/B-M			CEM IV/B			CEM I			CEM II/B-M			CEM IV/B		
		τ_o[a]	n_p[a]	R^{2a}	τ_o	n_p	R^2	τ_o	n_p	R^2	τ_o	n_p	R^2	τ_o	n_p	R^2	τ_o	n_p	R^2
100	1	0.75	7.40	0.98	–	–	–	–	–	–	0.04	3.40	0.99	0.21	5.00	0.99	0.15	3.80	0.97
	2	0.08	4.90	0.98	0.20	5.30	0.99	0.10	5.30	0.96	0.00	0.90	0.99	0.01	5.30	0.99	0.02	2.20	0.99
	3	0.01	5.40	0.99	0.02	6.60	0.99	0.01	6.40	0.99	0.00	1.00	0.99	0.01	2.10	0.99	0.01	1.70	0.99
40	1	1.24	8.00	0.99	0.99	8.70	0.96	–	–	–	0.00	4.20	0.99	0.01	5.50	0.99	0.28	2.80	0.97
	2	0.14	3.90	0.97	0.11	4.30	0.97	0.17	4.90	0.97	0.01	2.00	0.99	0.00	1.70	0.99	0.04	2.30	0.97
	3	0.05	3.10	0.95	0.02	4.70	0.98	0.06	4.60	0.98	0.00	1.50	0.95	0.00	1.40	0.99	0.01	1.70	0.99
20	1	–	–	–	–	–	–	–	–	–	0.19	4.50	0.99	0.33	2.50	0.99	–	–	–
	2	0.54	5.40	0.99	0.28	4.10	0.96	0.60	4.40	0.96	0.14	2.70	0.98	0.04	3.30	0.99	0.20	2.90	0.99
	3	0.16	3.60	0.98	0.09	3.20	0.95	0.20	3.70	0.98	0.06	2.10	0.99	0.01	1.70	0.99	0.09	2.70	0.99
10	1	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–	–
	2	1.25	3.20	0.97	0.70	4.20	0.99	1.32	4.60	0.96	0.28	3.80	0.99	0.20	1.10	0.96	0.48	3.90	0.99
	3	0.55	2.30	0.95	0.22	2.60	0.97	0.64	3.10	0.98	0.17	2.70	0.99	0.05	2.10	0.99	0.30	2.80	0.99

[a] τ_o: yield stress (Pa), n_p: plastic viscosity (mPa·s), R^2: correlation coefficient.

Table 4
Bleed capacity (%) of cement suspensions.

d_{max} (μm)	Without superplasticizer									With superplasticizer								
	W/C = 1			W/C = 2			W/C = 3			W/C = 1			W/C = 2			W/C = 3		
	CEM			CEM			CEM			CEM			CEM			CEM		
	I	II	IV	I	II	IV	I	II	IV	I	II	IV	I	II	IV	I	II	IV
100	29	16	27	59	50	53	68	64	65	39	16	17	61	47	43	71	67	60
40	18	18	17	47	44	42	62	60	56	26	29	7	55	46	44	69	67	59
20	2	4	0	25	27	20	49	45	40	0	2	1	37	35	26	46	49	42
10	0	2	1	7	14	30	39	38	43	0	2	2	11	19	35	42	38	47

capacity ⩽5% according to EN 12715, were obtained only for the fine-grained cements (d_{max} of 20 μm and 10 μm) and for W/C = 1, regardless of the use of superplasticizer. For the unstable suspensions, use of the superplasticizer usually resulted in increased bleed capacity but the opposite effect was obtained for a limited number of suspensions and, mostly, for the pozzolanic cements.

As expected, cement gradation had a measurable effect on bleeding. Pulverization from a d_{max} = 100 μm to d_{max} = 40 μm reduced bleed capacity by 2% to 20% for W/C = 2 and 3 and by 30% to 50% for W/C = 1. Pulverization from d_{max} = 100 μm to d_{max} = 10 μm reduced bleed capacity by 20–90% and yielded stable suspensions for W/C = 1. Cement type did not have a strong effect on bleeding since pulverization to microfine cement grain sizes yielded stable suspensions for all cement types at W/C = 1 and, in the range of unstable suspensions, bleed capacity values were not significantly different (average difference of 8%). Therefore, bleed capacity appears to be affected more drastically by the size than the composition (specific gravity) of cement grains.

7. Setting times

Setting times and early strength development of cement grouts are measured on the sediments produced after completion of bleeding. When the bleed capacity is small or negligible, the W/C ratio of the sediments is, practically, equal to the W/C ratio of the grout. This was confirmed in the present research for the cases of stable suspensions, that is, for suspensions of the fine-grained cements (d_{max} of 20 μm and 10 μm) at W/C = 1 prepared with and without superplasticizer. When bleeding is significant, measurements are necessarily made on sediments with a lower W/C ratio compared to that of the grout. It should be noted that the W/C ratio values of the sediments obtained after sedimentation in a large container may differ from those actually occurring in the pores of a grouted soil. The W/C ratio of the suspension sediments tested during this investigation ranged from 0.52 to 1.00, from 0.62 to 1.84 and from 0.64 to 1.75 for suspension W/C ratios of 1, 2 and 3, respectively. For the suspensions prepared at W/C = 2 and 3, with and without superplasticizer (unstable suspensions), the W/C ratio of the sediments increases with increasing cement fineness since it ranged from 0.62 to 1.02, from 0.71 to 1.15, from 1.13 to 1.65 and from 1.20 to 1.84 for cements with d_{max} equal to 100 μm, 40 μm, 20 μm and 10 μm, respectively.

The initial setting times of the suspension sediments ranged between approximately 4 and 12 h, while the final setting times ranged between approximately 6 and 21 h, with the exception of a small number of cases which had very long setting times. These cases are limited to suspensions of CEM IV/B cement at W/C = 2 or 3 and, generally, for gradations with d_{max} = 20 μm and 10 μm. These cases not considered, the elapsed time between initial and final setting ranged from 1.0 to 3.1 h, from 1.2 to 7.2 h and from 1.3 to 7.5 h for suspensions with W/C = 1, 2 and 3, respectively. The final setting time is of major importance to grouting practice, since a short time (<4 h) can damage equipment and a long time (>24 h) can delay execution of the works and reduce grouting efficiency [1]. Cement based grouts generally set within 4–24 h from preparation, depending on the additives used [32]. All results obtained for the final setting time of the suspension sediments, prepared with and without superplasticizer, are summarized in Table 5. The setting times obtained for the stable suspensions (d_{max} = 20 μm and 10 μm, at W/C = 1) range between 6 and 12 h and are in very good agreement with recorded times found in the literature [4]. In general, final setting times increase as the W/C ratio increases and, in most cases, when the superplasticizer is added to the suspension as also reported in available literature i.e. [9,12]. It can also be observed that cement type (pozzolan content) affects the setting times of the suspensions. CEM IV/B cement suspensions had, systematically, the highest initial and final setting times, compared to the other cement types used, regardless of cement fineness or superplasticizer presence. This behavior can be attributed to the more pozzolanic composition of CEM IV/B cement type (pozzolan content of 38% in comparison with 0% and 23.5% for CEM I and CEM II/B-M cement types, respectively) that affects the hydration process [9,33,34] and, possibly, to detrimental effects of the superplasticizer dosage.

8. Strength

As is the case with setting times, strength was measured for suspension sediments obtained by sedimentation in a large container, thus not simulating conditions in the pores of a grouted soil. The development of early strength was monitored for all suspensions using a pocket penetrometer, limiting the maximum obtainable value to 450 kPa. Typical results are presented in Fig. 7. In general, the early strength of the sediments obtained from the

Table 5
Final setting times (h) of cement sediments.

d_{max} (μm)	Without superplasticizer									With superplasticizer								
	W/C = 1			W/C = 2			W/C = 3			W/C = 1			W/C = 2			W/C = 3		
	CEM			CEM			CEM			CEM			CEM			CEM		
	I	II	IV	I	II	IV	I	II	IV	I	II	IV	I	II	IV	I	II	IV
100	8.5	14.5	16.7	8.9	18.1	21.1	11.4	17.2	25.5	8.6	8.3	11.1	8.5	8.0	16.0	9.1	13.8	16.0
40	5.7	6.8	8.7	6.5	8.9	12.7	7.6	10.2	22.2	7.8	9.8	21.0	8.1	11.0	13.3	7.9	12.2	18.3
20	6.4	8.0	9.5	13.1	14.1	46.2	13.5	16.7	46.7	8.4	8.2	10.0	9.2	11.7	>48	10.7	19.0	26.5
10	7.7	5.9	10.8	17.9	14.3	>48	16.7	15.0	>48	9.8	5.7	12.3	14.3	7.9	>48	17.3	8.2	>48

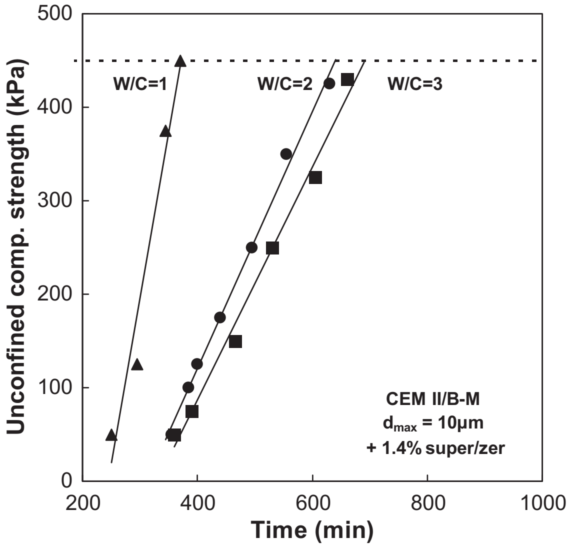

Fig. 7. Typical early strength development of grout sediments.

cement suspensions is a linear function of time. A review of the available results indicates that the early strength of the cement suspensions is affected by the W/C ratio of the suspensions as well as by the cement type and fineness. Suspensions with W/C = 1, 2 and 3 approach a strength value of 450 kPa within a time period of 5.4–17.8 h, of 6.9–49.3 h and 7.4–108.8 h, respectively. These ranges are practically independent of the presence of superplasticizer in the suspensions. Furthermore, as typically shown in Fig. 8, for similar sediment W/C ratios, the characteristic strength value of 450 kPa is approached faster as the cement fineness increases. Similar observations have been reported by Schwarz and Krizek [6]. In terms of cement type, the time needed for approaching a strength value of 450 kPa, ranged from 5.4 to 22.9 h, from 7.1 to 29.1 h and from 8.2 to 49.3 h for CEM I, CEM II/B-M and CEM IV/B, respectively, indicating that early strength depends on the percentage of clinker used for cement production (90%, 63%, 58%, respectively).

The unconfined compression strength of the cement sediments increased with increasing curing time, increasing cement fineness and decreasing W/C ratio. The 3-day strength was, generally, more than 50% of the 28-day strength, and the strength after curing for 28 days ranged widely from as low as 1.5 MPa to as high as

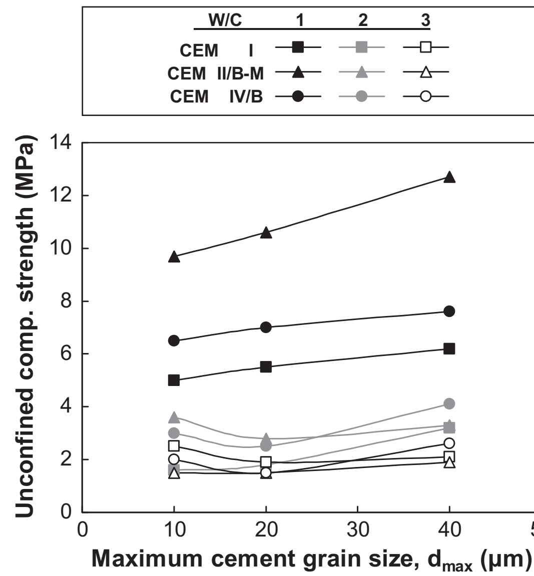

Fig. 9. Unconfined compression strength of cement sediments with superplasticizer.

16.4 MPa. As expected, the W/C ratio of the suspensions has a significant effect on the strength of the sediments. The results for cement sediments cured for 28 days and obtained from suspensions with W/C = 1, 2 and 3 prepared with superplasticizer are shown in Fig. 9. It can be observed that increase of the W/C ratio from 1 to 2 and 3 resulted in a decrease of the unconfined compression strength of the sediments by a factor of 2 to 4. Numerous similar observations have been reported i.e. [35–38]. The results shown in Fig. 9 indicate erroneously that increased fineness of the cements results in decreased strength. This is attributed to the fact that the results shown do not reflect the effect of sediment W/C ratio. The effect of cement fineness on sediment strength, for similar sediment W/C ratios, is presented in Fig. 10. It can be observed that the increase of cement fineness from d_{max} = 100 μm to d_{max} = 20 μm and 10 μm causes an increase of the unconfined compression strength of cement sediments by a factor of 2–3. This observation is in good agreement with reports by Lapasin et al. [39], Schwarz and Krizek [6], Saleh et al. [40] and Shibata [36], and can be attributed to the higher hydration activity of finer

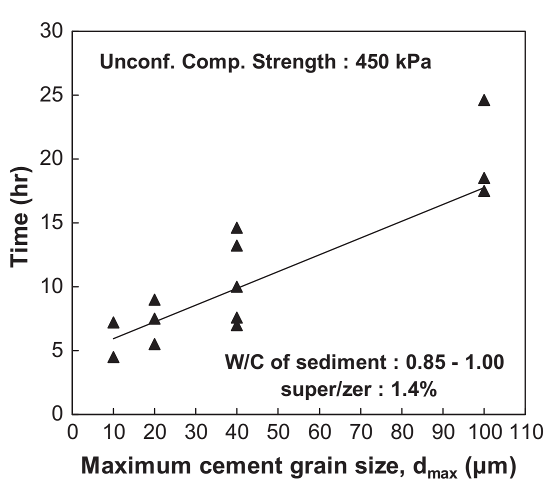

Fig. 8. Effect of cement fineness on early strength development.

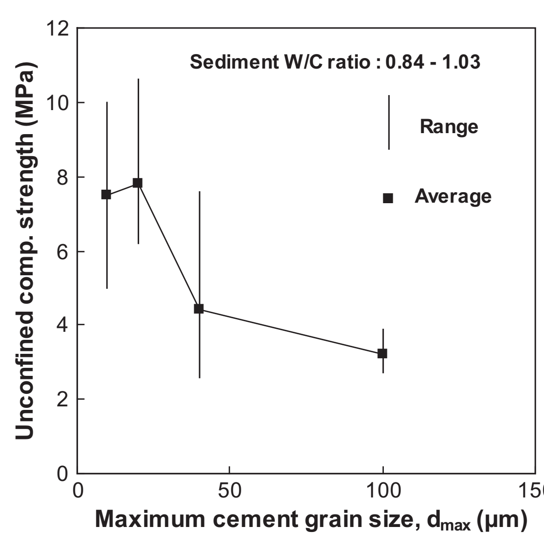

Fig. 10. Effect of cement fineness on sediment strength.

grains and more efficient hydration process as well as to the fact that smaller grains also means smaller spaces between the grains that could increase compressive strength due to reduced flaw sizes.

Cement type seems to affect the unconfined compression strength of the sediments. As shown in Fig. 9, and especially for the sediments of fine-grained cement suspensions (d_{max} = 10 μm and 20 μm) the highest strength values were obtained for the CEM II/B-M cement containing 23.5% of pozzolan. Similar observations, where microfine composite or pozzolanic cements exhibited higher unconfined compression strength than ordinary Portland cement, have been reported [6,41].

9. Groutability

The results of the groutability tests were characterized as "satisfactory" when the predetermined quantity of grout (two void volumes of the sand column) were injected with a grouting pressure not exceeding 200 kPa, as "questionable" when either the volume of injected grout was less than the predetermined quantity or grout penetration was less than the length of the sand column (400 mm), and as "impossible" when grout penetration was very small under the maximum applied pressure (200 kPa). A preliminary evaluation of groutability was also made using as criteria the "groutability ratios" [5] which are defined as $N_1 = (D_{15})_{soil:} / (D_{85})_{grout}$ and $N_2 = (D_{10})_{soil:} / (D_{95})_{grout}$. Grouting is considered possible if $N_1 > 25$ and $N_2 > 11$ and not possible if $N_1 < 11$ or $N_2 < 6$. If $N_1 \geqslant 50$, satisfactory permeation can be achieved. Presented in Fig. 11 are the results obtained from the groutability tests together with the corresponding groutability ratio values, N_1, for all cements, W/C ratios and sand gradations used in this investigation. For different cement types of the same d_{max} injected into a specific sand, small differences are obtained for the groutability ratio values which are due to differences in the d_{85} values of the cements. Results for the N_2 ratio are not presented since, for the cases investigated, predictions are less conservative than those obtained by applying the N_1 ratio. It can be observed that groutability was "satisfactory" in coarse sands, regardless of the W/C ratio of the suspensions. Groutability in medium sands was, generally, "satisfactory" for cement suspensions with groutability ratios $N_1 > 25$. The medium-to-fine sands were grouted "satisfactorily" with cement suspensions having, generally, $N_1 > 25$ and W/C = 2 or 3. Penetration in fine sands was negligible for any cement suspension used. The effect of cement type on groutability was not significant. In terms of observed performance, cements types I and II/B-M are rated as about equivalent and slightly superior to cement type IV/B.

For tests with the medium/fine sand (d_{15} = 0.34 mm) eleven different cement gradations were tested, with groutability ratio, N_1, values ranging between 9 and 57 indicating the full range of possible predictions on the basis of this ratio. The performance of the three suspensions of the original cements having nominal d_{max} of 100 μm and yielding N_1 values between 9 and 12 was accurately predicted by the N_1 criterion. Suspensions of the three different cements with nominal d_{max} of 40 μm have corresponding N_1 values 16, 19 and 22 which are in the range of 11–25 where the N_1 criterion suggests some positive possibility for grouting. The experimental results indicate satisfactory injection only for suspensions of the type I cement where a relatively high N_1 value (22) was combined with low apparent viscosity values (1.6 to 4.4 mPa s) at preparation. Grouting with suspensions having nominal d_{max} of 20 μm and 10 μm should be possible according to the groutability criterion since $31 \leqslant N_1 \leqslant 57$. However, this was not confirmed for the cases of suspensions with W/C = 1 which had an apparent viscosity value at the time of preparation ranging from 23 to 180 mPa s. Conditions were improved when the W/C ratio was reduced to 2 yielding apparent viscosity values between 5 and 10 mPa s and even further improved for suspensions with W/C = 3 (apparent viscosity between 2 and 5.4 mPa s). For these cases, the experimental observations indicated "satisfactory" injection for viscosity values up to 5 mPa s, "unsatisfactory" for a viscosity value of 10 mPa s and "questionable" for intermediate values.

This information indicates that predictions based on the groutability ratio values and observations from laboratory injection tests are mostly in agreement. However, for a number of cases, the groutability ratios yield rather optimistic predictions which are not confirmed experimentally. Similar prediction inefficiency has been documented in the available literature i.e. [42–44]. There are cases of cement suspensions with groutability ratio values greater than 25 or even 50, where the observed groutability was either

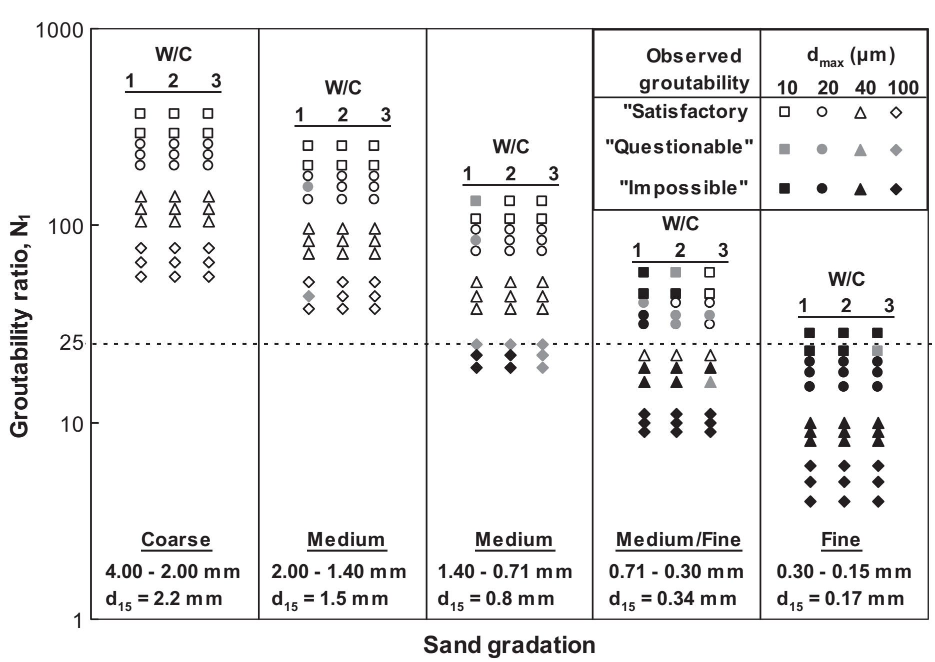

Fig. 11. Groutability of cement suspensions.

"questionable" or "impossible". This behavior is attributed to the fact that groutability ratios are based solely on characteristic grain sizes of grout and soil and do not take into consideration factors, such as W/C ratio and viscosity, which have an effect on groutability.

10. Effectiveness

A rather limited number of unconfined compression tests was conducted on grouted sand specimens after curing for 28 days. The effect of cement type and gradation, grout W/C ratio and sand gradation can be evaluated in terms of the results shown in Fig. 12. In terms of cement type, the available information cannot establish the superiority of any one of the cements, as shown in Fig. 12a. There are, however, strong indications that the unconfined compression strength of the grouted sands decreases with increasing W/C ratio and increases with increasing cement fineness, as shown in Fig. 12b for a sand with d_{15} = 0.8 mm grouted with II/B-M cement suspensions having W/C = 1, 2 and 3. The unconfined compression strength ranged from 4.0 to 11.2 MPa, from 0.5 to 1.2 MPa and from 0.5 to 1.0 MPa, for W/C = 1, 2 and 3, respectively. The effect of cement fineness is most pronounced for the thicker cement suspensions (W/C = 1) and is negligible (±20%) for W/C = 3. Similar observations on the effect of W/C ratio and cement fineness on unconfined compression strength and comparisons between ordinary Portland and microfine cement grouts have been reported in the available literature [42,43] and can be attributed to the fact that grouting with a higher solids content and/or finer cement gradations results in better filling of the soil voids and yields improved cementation of the sand grains. The unconfined compression strength values (range from 6.0 to 11.0 MPa) obtained by grouting with the microfine cement suspensions (d_{max} = 10 and 20 μm) having W/C = 1, can be considered as very satisfactory for stabilization applications. Finally, as shown in Fig. 12c, sand gradation has a significant effect on grouted sand strength. It can be observed that an increase of unconfined compression strength by a factor of 3 was obtained as the d_{15} of the grouted sand was reduced from 2.2 mm to 0.34 mm. This observation is attributed to the increased number of grain-to-grain contact points in a finer soil and, as a result, to the increased number of points available for cementation [42].

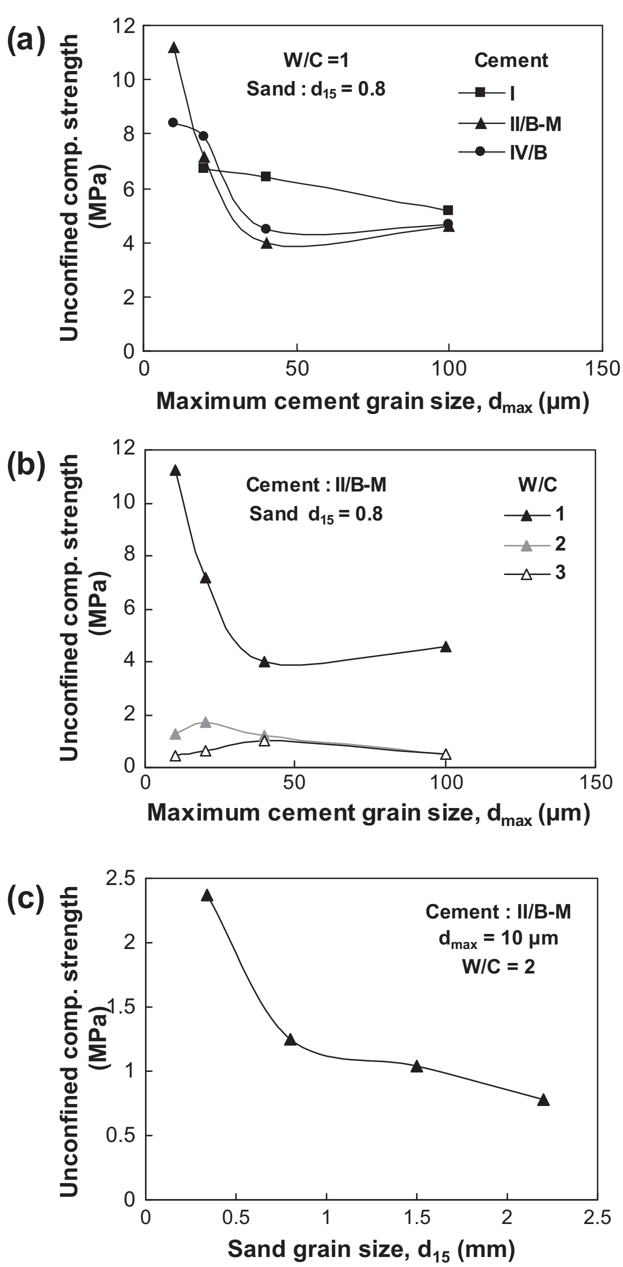

Fig. 12. Unconfined compression strength of grouted sands; effect of (a) cement type, (b) cement gradation and grout W/C ratio and (c) sand gradation.

11. Conclusions

Based on the results obtained and the observations made during this experimental investigation and within the limitations of the range of parameters investigated, the following conclusions can be advanced:

1. Pulverization of the ordinary cements to produce microfine cements (a) has a detrimental effect on apparent viscosity which increased by an average of 500%, (b) improves bleeding, (c) has a positive effect on setting times, rate of early strength development and 28-day unconfined compression strength, (d) extends the range of groutable sands to "medium-to-fine" and (e) yields very satisfactory unconfined compression strength of grouted sands.
2. The detrimental effect of pulverization on apparent viscosity is negated by adding a superplasticizer, based on polycarboxylate chemistry, to the suspensions.
3. All suspensions, with or without superplasticizer, can be considered to behave as Bingham fluids with model parameter values for superplasticized suspensions reduced by a factor of up to 5 compared to values obtained when no superplasticizer is used.
4. Increased pozzolan content from 0% (cement I) to 23.5% (cement II/B-M) and 38% (cement IV/B) has no effect on bleed capacity, groutability and effectiveness.
5. In terms of lower apparent viscosity and Bingham model parameter values, as well as 28-day unconfined compression strength, the best results are obtained for the intermediate (23.5%) pozzolan content which also yielded the lowest setting times and intermediate early strength development characteristics.
6. Groutability predictions, based on the groutability ratios, are mostly in agreement with experimental observations. Cases of prediction inefficiency can be attributed to the fact that groutability ratios do not take into consideration factors such as W/C ratio and viscosity.
7. The unconfined compression strength of grouted sands increases with decreasing W/C ratio of the suspension and with increasing cement fineness. Strength values range up to about 11 MPa and are considered very satisfactory for stabilization applications.

Acknowledgments

The research effort reported herein is part of the Research Project (PENED-03ED527) which was co-financed by the E.U.-European Social Fund (75%) and the Greek Ministry of Development – GSRT (25%). The contribution of TITAN Cement Company S.A. was substantial for the selection, chemical analysis, pulverization and grain size analysis of the cements.

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