Fundamental Observations on Cement Based Grouts (2): Microfine Cements and The Cemill^R Process

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Abstract

This paper describes the results of an extensive laboratory testing program conducted on two groups of microfine mixes, with and without bentonite. The aim of the research was to define the rheology and stability of the microfine mixes, their penetration capability and the permeability and strength characteristics of the treated soil. The relationship between the grain size of the grouts and the soil permeated by soffusion was also studied.

Introduction

During the last 40 years, various chemical grouts have been developed to penetrate finely fissured rocks or medium-fine sands. Recently, the greater attention devoted to the environmental compatibility of grouts has led to a critical and cautious attitude in their selection. Not coincidentally, there is at the same time a renewed interest in cement based grouts and considerable attention is now being devoted to attempts to increase their penetrability. De Paoli et al. (1992) have described results obtained on the rheologic properties of mixes using cements of traditional (or conventional) grain size. This companion paper reviews the possibilities offered by cementitious grouts using materials of microfine grain size.

Injection By Soffusion

Researches have featured attempts to isolate the parameters which regulate the permeation of suspensions

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with extremely fine solid contents into a granular skeleton (soffusion). Most attention has been devoted to the geometric and volumetric aspects of the phenomenon, relegating to second place the rheologic properties of the grout: in fact, permeation is controlled by the size of the particles more than by viscosity and cohesion. The conditions necessary for soffusion can be divided into a) geometric (the grain size must be smaller than the pore size) and b) hydraulic (the hydraulic gradient must be sufficient to cause the movement of the particles). Considering the pressures normally involved in grouting, the analysis of the second condition is invariably less important than the first.

The size of the pores of granular soils, and the aperture of rock fissures are dominant controls over groutability. While the geometry of a fissure is relatively simple to model, the pore system of a loose soil is complex. The grain size characteristics may be evaluated based on parameters like the effective diameter, the degree of uniformity and the specific surface of the grains. An appraisal of the dimensional distribution of the pores may be made from the grain size curve of the soil and from the shape of the grains using the formulae of Kozeny (1953) or, experimentally, by means of mercury porosimetry.

Originating from Terzaghi's criterion for filter sizing, the study of the geometric conditions of soffusion has led to the definition of the degree of groutability (N) based on the ratio between the grain size of the soil and of the solids of the grout. Mitchell (1981) defined the following:

$$N = (D_{15})\text{soil}/(D_{85})\text{grout} \qquad (1)$$

$$N_c = (D_{10})\text{soil}/(D_{95})\text{grout} \qquad (2)$$

with $N<11$ or $N_c<6$ injection would be impossible, while good results should be obtained with $N>24$ or $N_c>11$. However, according to Cambefort (1967), a more logical criterion of injectability should consider the dimensions of the voids as compared to those of the grains of the grout. Expressing the hydraulic radius of the mean interstitial section as a function of the porosity and specific surface, and using the Kozeny formulae to determine permeability, he derived the relationship:

$$D \leq C \quad .K^{1/2} \qquad (3)$$

where D is the average diameter of the grains of the suspension, C is a constant, and K is the permeability coefficient. More recenty, Arenzana et al. (1989) have indicated, with specific reference to the grouting of very thin microfine cement mixes (C/W = 0.25), the

	grain size (μmm)					
	D 95	D 85	D 60	D 50	D 15	D 10
CEMILL® 6	15.0	9.0	6.0	5.0	1.3	0.9
CEMILL® 9	9.0	5.5	3.5	2.5	0.6	0.4
CEMILL® 12	6.0	4.0	3.0	2.2	0.4	0.3
ONODA MC-500	8.0	60.0	4.5	4.0	2.5	2.0
Portland 525	40.0	22.0	11.0	8.0	2.5	2.0
bentonite	60.0	40.0	15.0	10.0	1.7	1.2

ⓐⓑⓒ sands for injection tests
ⓐ $\gamma = \gamma_{max} = 1.713$ g/cm^3
ⓑ $\gamma = \gamma_{max} = 1.701$ g/cm^3
ⓒ $\gamma = \gamma_{max} = 1.690$ g/cm^3

ⓓ bentonite
ⓔ Portland 525 cement
ⓕ ONODA MC-500 cement
ⓖⓗⓘ CEMILL® mixes

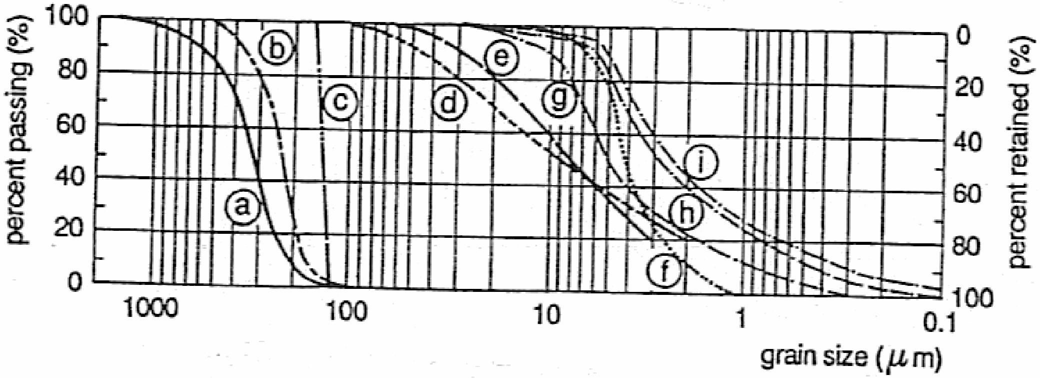

Figure 1. Grain size distribution curves for sands, dry materials and grouts.

following permeation conditions apply at a pressure of 70 kPa:

$$D_{10sand} \geq 0.15 \text{ mm} \qquad (4)$$
$$R_H = n/\ (1-n)\ S_o \cdot \gamma_d \geq 2\mu m \qquad (5)$$

where R_H (hydraulic radius) is the ratio between the surface available for the flow and the wet parameter; n, γ_d and S_o are the porosity, dry density and specific surface of the sand, respectively.

Microfine Cements

The traditional (or conventional, ordinary) cements normally have a specific Blaine surface of between 2500 and 5000 cm^2/g and grain sizes which at best range from 63 to 0.1 μm, as shown in Figure 1 (curve e). Microfine cements are produced by either dry or wet processes.

In the conventional dry method, microfine cements are manufactured by further refining the particles beyond the usual industrial limit (by dry milling) or fractionating (by ventilation) the cements available on the market, in order to draw from them only the target fineness fraction. With the wet method, it is possible to exploit the decantation principle under which particles of equal density fall, in a liquid, with a speed proportional to their diameter. To produce dry cement in this way, an inert liquid must be used (e.g. alcohol, liquid gas) which must afterwards be eliminated by means of filtration or evaporation. If water is used, microfine cement based grouts as opposed to dry microfine cements are produced.

The microfine cements must be used with great care, respecting effects resulting from the extreme fineness of their grains. In dry form, they tend to compact due to their sensitivity to humidity and electrostatic phenomena (i.e. a problem of transport and storage). When mixed with water, they tend to flocculate, losing the fineness that is their fundamental characteristic. In the preparation of grout mixes, this problem is partially solved by the use of dispersive additives which, by making the potential of the particles as negative as possible, prevent their aggregation. Figure 2 illustrates the phenomenon, comparing the granulometric curves, obtained with diffractometer, of the same microfine cement in aqueous suspension, with or without an additive.

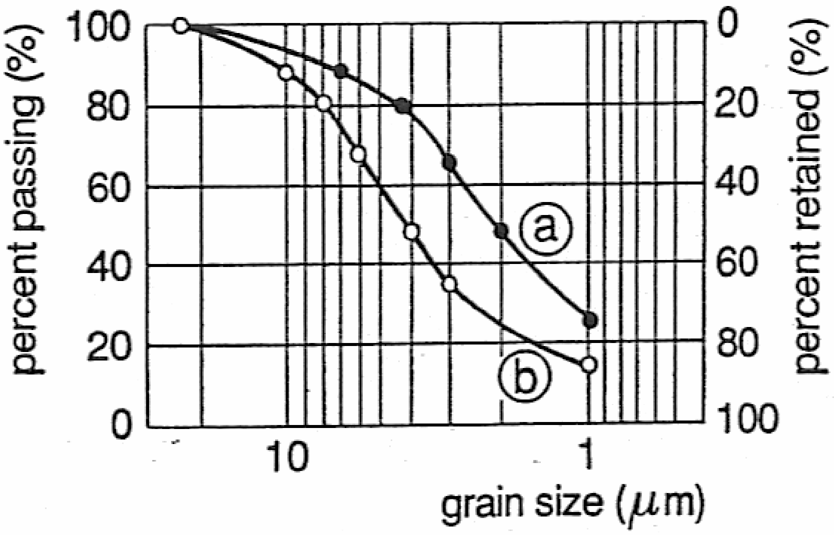

Figure 2. Grain size distribution curves of the same microfine cement in aqueous suspension (a) with and (b) without, dispersive agent.

CEMILL^{R} Process

The initial goals of the study were as follows: a) to evolve an on site manufacturing process to provide microfine grouts; b) to use, as the starting material, conventional cements, in order to be independent of the local market availability of microfine cements. (At present only available in a limited number of industrialized countries, and often at fairly high cost); c) to overcome the limits of pure decantation which permit only the manufacture of very diluted grouts and implies the difficult disposal of the coarse, rejected portions; and d) to make possible the production of not only unstable mixes (water, cement, possibly dispersive agent), but also stable grouts using bentonite, and therefore unobtainable by decantation.

The achievement of these goals has been made possible by the development of a special colloidal wet refiner which has a dual function: a) it imparts a very strong dispersive action to the cement used in the grout. In this way the effectiveness of the microfine portion already contained is safeguarded even if dispersive agents are not used; b) it carries out a progressive refining process of the coarse portion, until the desired fineness is reached, without requiring its physical elimination as in the case of decantation.

The energy expended in this process, and therefore the overall cost, depends also on the option of using bentonite. If the process is to provide unstable mixes (CEMILL-I, Figure 3) the refiner's work is facilitated by the action of a continuous separator which selects the fine fraction already available. In this case, the use of a dispersive agent (i.e. naphthalene sulfonate) will help. If, however, bentonite is used to give stable grouts (CEMILL-S, Figure 3b), the separator is no longer capable of performing its function and the entire process is carried out by the colloidal refiner. Curve (e) of Figure 1 shows the Portland 525 (Type III) cement used to manufacture the starting mix; curves (g), (h) and (i) show the grain size distribution of three CEMILL mixes, prepared with different refining times (6, 9, 12 minutes). Monitoring of the grain size of the cements has mainly been carried out with mercury porosimetry on grout samples, after eliminating the aqueous phase by means of washing with organic solvent and subsequent filtrations (and elimination of the solvent residues). It is important to stress that the grain size curves in Figure 1 do not therefore refer to anhydrous products but to cement particles effectively present in the mix and which have already potentially undergone the effects of aggregation or swelling due to hydration. Figure 1 also shows the grain size curve of the MC-500 cement of the Onoda Cement Corporation, the first producer to make microcements commercially available (Clarke,1984)

Laboratory Tests on CEMILL^R Mixes

Laboratory tests were performed to characterize the two groups of mixes (CEMILL-I and CEMILL-S) rheologically and with respect to stability, penetrability, strengthening and waterproofing capacity.

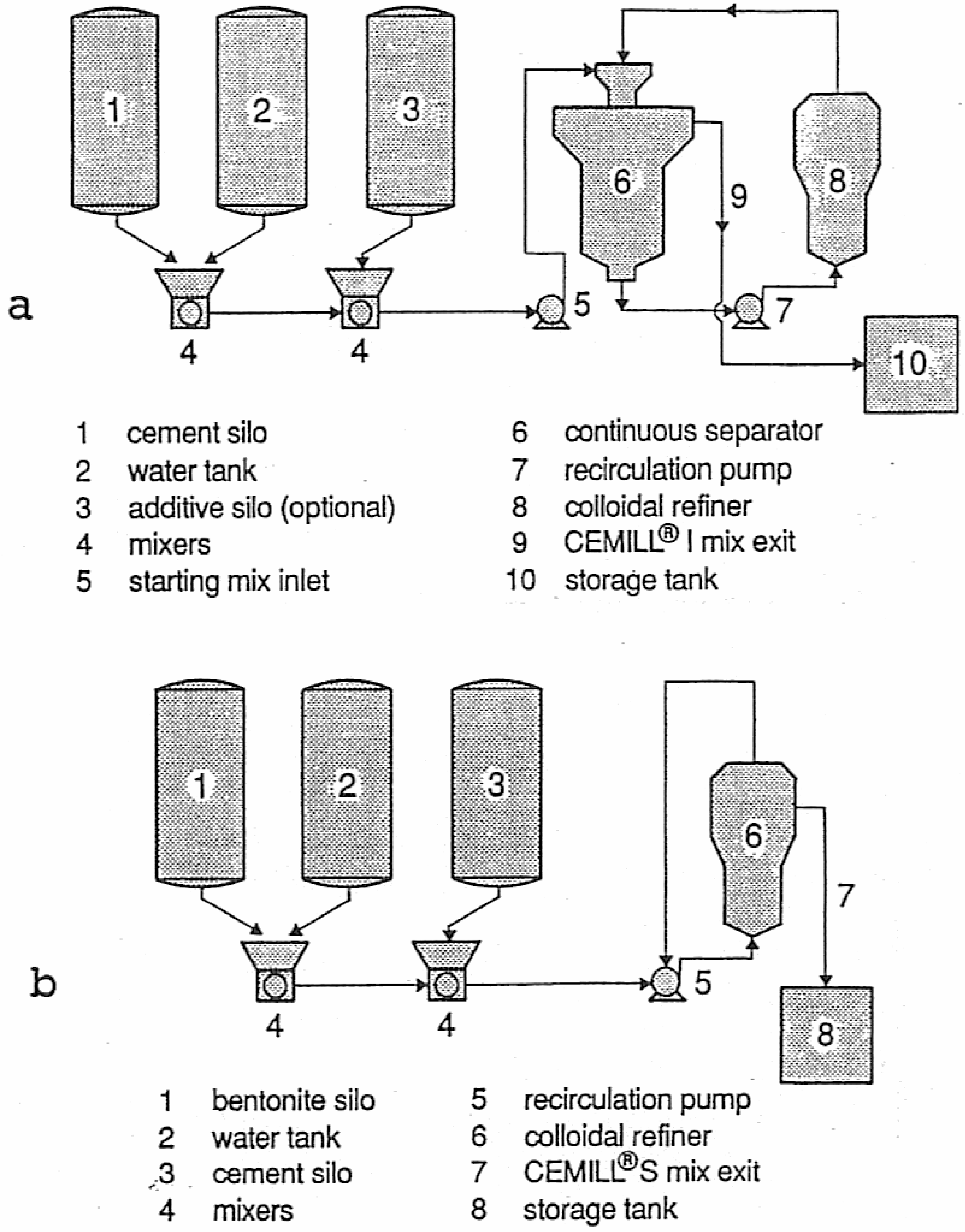

Figure 3. Layout of production plants for (a) CEMILL-I (unstable grouts) and (b) CEMILL-S (stable grouts).

The two groups differ essentially in their stability (bleed) characteristics. For the CEMILL-I mixes, which are unstable as they do not contain bentonite, the C/W range was extended from 0.2 to 1. The CEMILL-S grouts, which are stable as they contain bentonite, were studied in the C/W range of 0.2 to 0.5 (beyond this value the addition of bentonite no longer has much relevance).

The bentonite content in the CEMILL-S grouts was maintained constant, with a weight ratio to water of 4%, and was always mixed with water before the addition of cement.

The time of mixing-refining was varied between 6 and 12 minutes, controlling the subsequent grain size distribution. The testing methods for viscosity, cohesion, bleed, pressure filtration, strength and permeability testing were described by De Paoli et al. (1992). The results now described relate tomixes refined for 6 minutes. The stability to bleed of the two groups of mixes is summarized in Figure 4a. The CEMILL-S mixes were stable (no bleeding), whereas the CEMILL-I mixes showed a bleed capacity between 60 and 0.4% at 8 hours, depending on the C/W ratio. This is decidedly lower than that of the corresponding mixes manufactured with cements with traditional grain sizes. This improved stability to decantation is attributable to the fineness and dispersion of the particles, which are capable of sedimenting singly and slowly.

The rheological characteristics were measured on freshly manufactured mixes and after 30, 60, and 90 minues of aging. The cohesion (Figure 4b) of CEMILL-I mixes remained practically zero in the 90 minutes of observation. The initial cohesion of the CEMILL-S mixes, although rather higher than that of CEMILL-I, did remain relatively low, varying between 6 and 12 Pa. The apparent viscosity, Figure 4c, measured on the two groups of mixes, was between 2 and 6cP (CEMILL-I) and between 10 and 18cP (CEMILL-S).

The characteristics of stability to pressure filtration are correlated, in terms of pressure filtration coefficient k_{pf} (De Paoli et al., 1992) with the initial cohesion in Figure 5. The results may be compared with the correlation supplied by De Paoli et al. (1992) for the mixes using conventional cements. The CEMILL-I grouts presented pressure filtration characteristics as good as those of conventional grouts stabilized with bentonite. The CEMILL-S mixes were, on the contrary, in the field of the MISTRA mixes (De Paoli et al. 1992).

Short-term shear strength characteristics were measured with Solexpert Cohesimeter and Vane scissometric equpment up to their instrument limit of 4 and 180 kPa respectively (Figure 6). Due to their inherent instability, the CEMILL-I mixes generate by bleeding mixes with final C/W ratios much higher than the initial ones, so that mixes with very different initial C/W ratios end up giving similar high rigidity and shear strength. The CEMILL-S mixes, however, preserve the

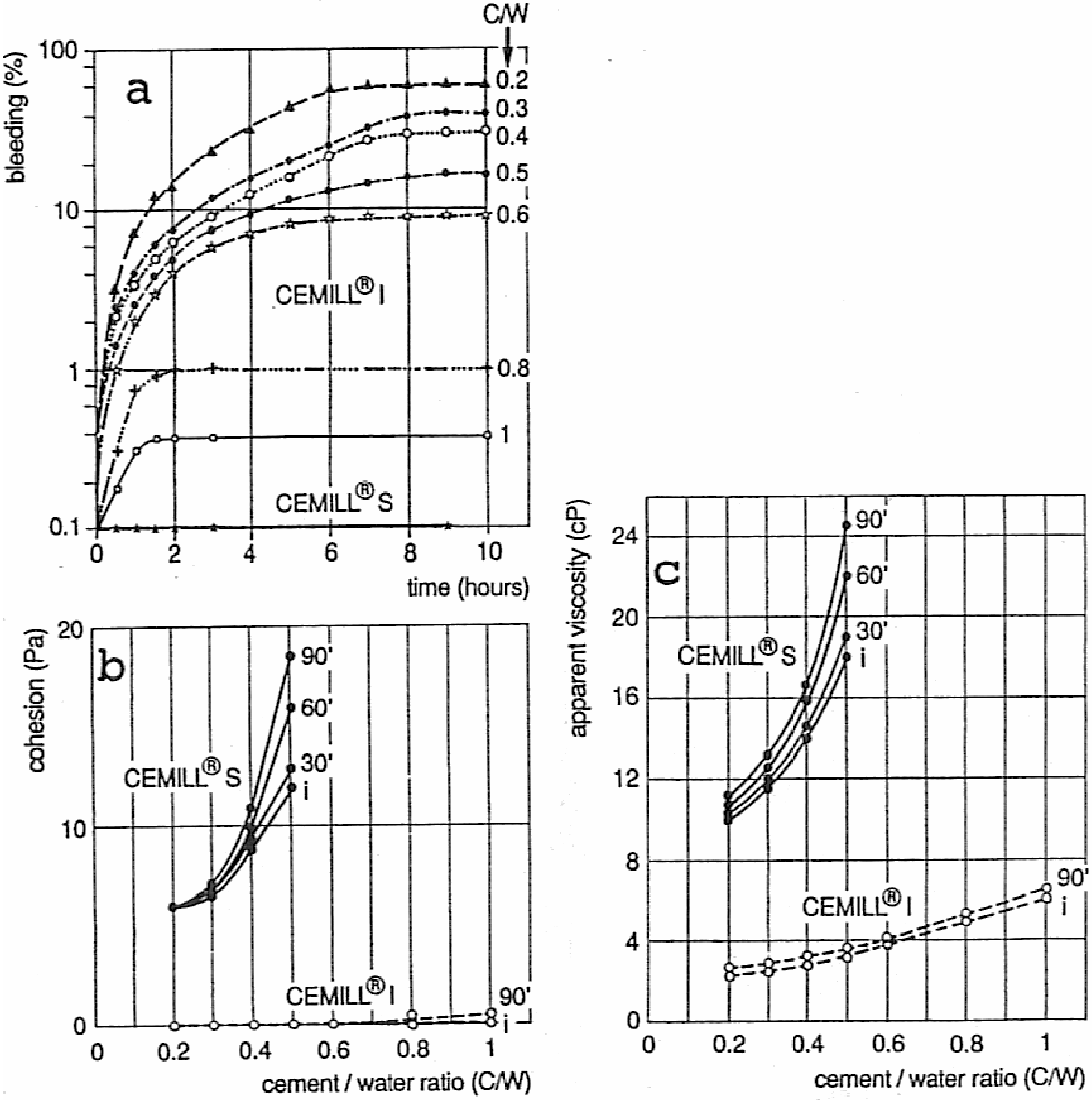

Figure 4. CEMILL^R mixes: rheology and stability as a function of cement content.

initial C/W ratio unaltered and so presented a wider range of stiffening times. The initial setting time, calculated as the time necessary to surpass the conventional threshold, τ = 18 kPa, exceded 24 hours only in the case of CEMILL-S with C/W=0.3. It was therefore of the same order of magnitude as that of conventional cement mixes.

The groutability study of CEMILL mixes was conducted using the mould injection method (De Paoli et al., 1992). The sample was prepared by inserting, compacting and saturating sand with preselected grain size in a flexible PVC tube (H=500mm, d=40mm), equipped at the ends with filters and expansion shutters to prevent swelling during the subsequent grouting phase.

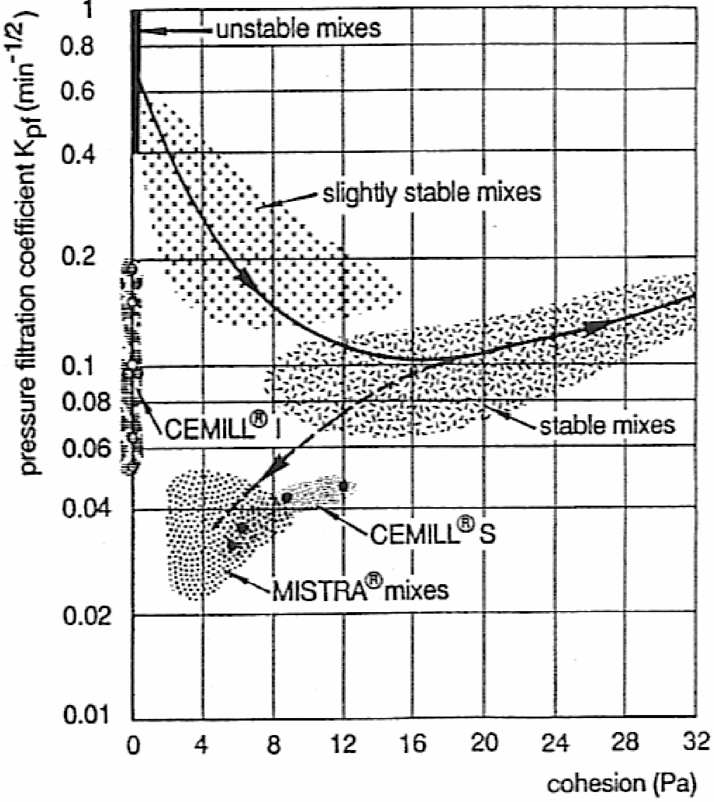

Figure 5. Relationship between stability under pressure and initial cohesion for CEMILL^R and other mixes prepared with cements with traditional fineness.

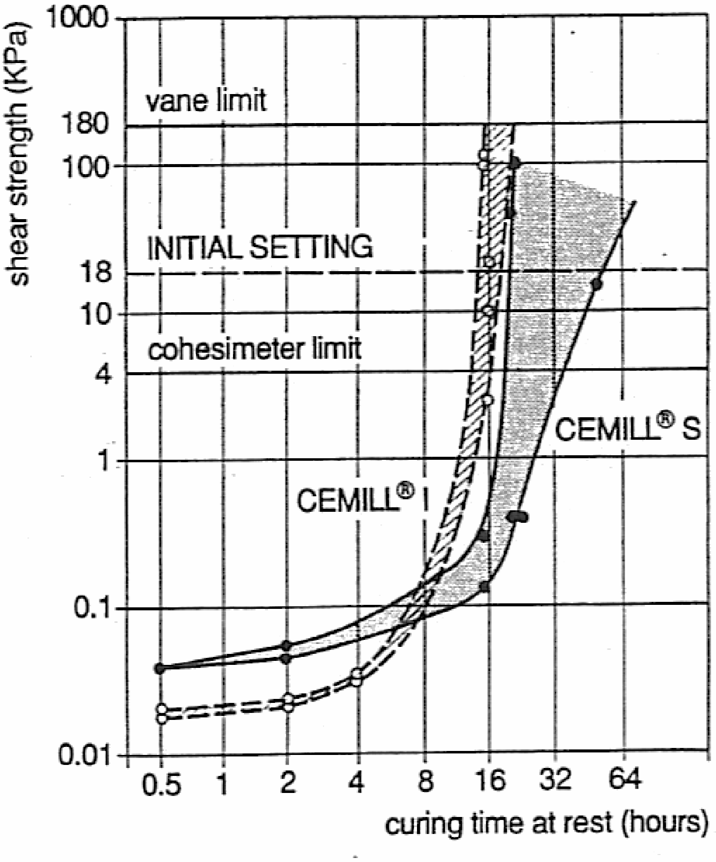

Figure 6. CEMILL^R mixes: shear strength development with time.

Different tests of this type have been performed using sands with the grain size shown in Figure 1. The results may be summarized as follows: 1) sand-a is permeated by all CEMILL mixes; sand-b is, in the majority of cases, also permeated by all CEMILL mixes, but there have been cases of incomplete permeation; 3) sand-c is sporadically grouted only by CEMILL mixes with type-i grain size and with C/W ratio not over 0.3.

The method of mould preparation depends on the sand used and the procedures adopted, and so it leaves wide margins of doubt and makes it difficult to reproduce tests, particularly when these are performed by different laboratories. To better evaluate the relationship between grout and grouted body, an instrument has been developed (Figure 7) to permit the injection of standard porous filters. The test consists of injecting the mix (under pressure, always below 0.5 MPa) through the filters, verifying whether the mix is retained or passing. These filters, commercially available (Filtri

filter no.	permeability (m/s)		grain size (μm)					porosimetry (μm)							specific surface cm²/g	retaining capacity (μm)
	theoretical Hazen (C = 1.45)	experim. (permeam.)	D 95	D 60	D 15	D 10	U	theoretical (Kozeny) D 80	D 50	D 30	experim.(Hg porosimetry) D 95	D 85	D 15	D10		
07	$5.9 \cdot 10^{-3}$	$3.8 \cdot 10^{-3}$	1500	900	700	640	1.41	300	240	150	380	300	170	160	28	70
06	$2.3 \cdot 10^{-3}$	$8.3 \cdot 10^{-4}$	750	620	450	400	1.55	160	133	90	360	260	130	124	37	60
04	$7.7 \cdot 10^{-4}$	$4.5 \cdot 10^{-4}$	700	480	250	230	2.09	110	90	60	300	140	70	64	56	40
01	$2.8 \cdot 10^{-4}$	$1.6 \cdot 10^{-4}$	400	230	160	140	1.64	58	49	32	120	64	46	44	111	10
005	$1.4 \cdot 10^{-4}$	$9.5 \cdot 10^{-5}$	180	120	110	100	1.20	35	25	18	90	46	32	30	125	5

a

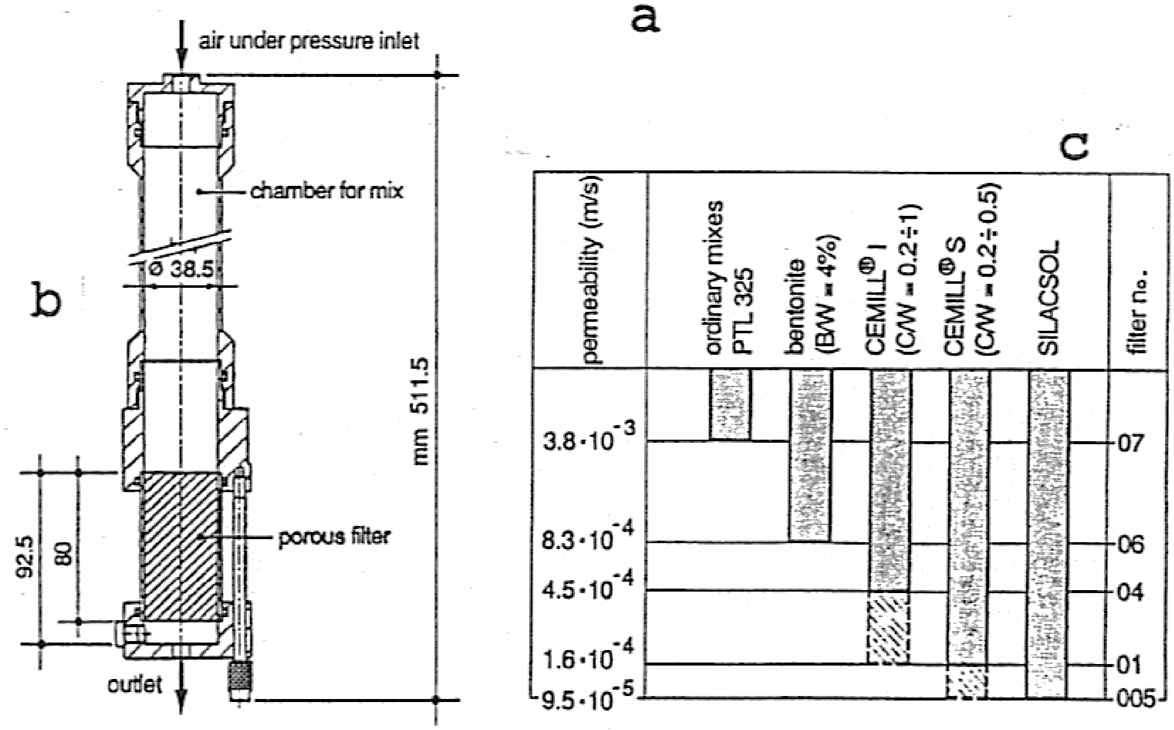

Figure 7. Injection test details: a) porous stone filter characteristics, b) apparatus, and c) penetrability limit of different mixes into filters.

Fink S.a.S., Milano), incorporate sands of different predetermined grain size, cemented with epoxy resin. These are standard filters, and so are independent of the sand used and the technician's skill. The filters are characterized in particular by a standardized pore diameter distribution, measurable, for example, with a mercury porosimeter. The grain sizes, the pore distribution, the theoretical and experimental permeability and the specific surface (calculated assuming spherical grains) of the filters used are illustrated in Figure 7a. Test results are summarized in Figure 7c. Filter 07 was permeated by ordinary mixes using Portland cement; filter 06 was not permeated by these mixes but by 4% bentonite slurry and by Type III cement mixes; filter 04 was permeated by all CEMILL mixes; filter 01 was permeated by CEMILL-S and, only for low cement contents, by CEMILL-I-12; filter 005 was permeated by silicate mixes (Tornaghi et al., 1988) and only by CEMILL-S-12 with low cement content.

Further, the injection of sand moulds has been used to obtain samples in order to evaluate the strength and permeability of sand treated with CEMILL mixes. In compacted sand (type a of Figure 1), CEMILL-I mixes with C/W ratio = 0.2 to 1 and CEMILL-S with C/W = 0.2 to 0.5 have been injected, at pressures always below 0.5 MPa. After injection, the sample was maintained in a vertical position until the grout had set, then dismantled and cut into sections (H = 80 mm) for compression tests (ASTM-

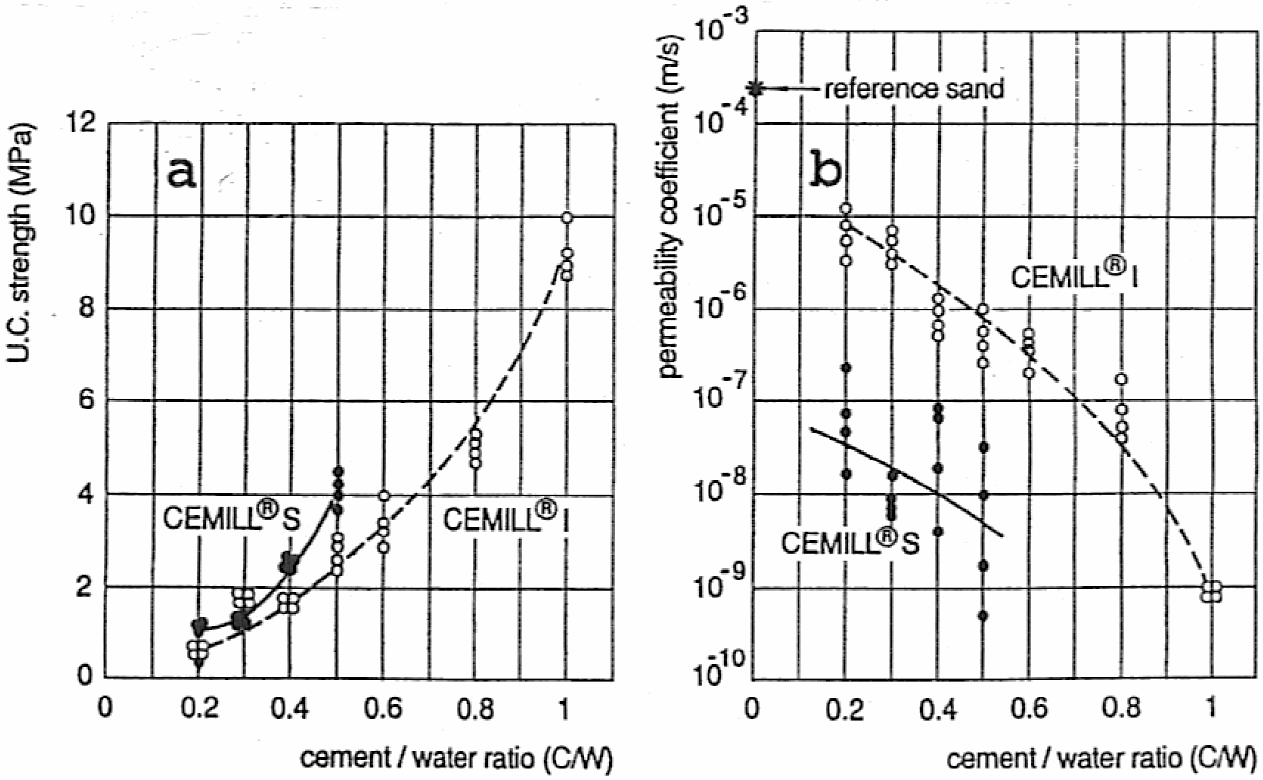

Figure 8. CEMILL^R mixes: (a) unconfined compressive strength and (b) permeability.

C39) and permeability (in triaxial cell under constant head) tests. In Figures 8a and 8b the unconfined compressive strength and peremability values are shown as a function of the C/W ratio of the grout. The compressive strengths obtained at 28 days varied as a function of the C/W ratio from 1 to 4 MPa for those treated with CEMILL-S, and between 0.65 and 9 MPa for those treated with CEMILL-I. For the same samples the permeability coefficient, measured at 28 days, was reduced from the original $2x10^{-4}$ m/s to 10^{-9} m/s. With equal cement content, the stable mixes (CEMILL-S) show a higher strength and lower permeability than the CEMILL-I grouts.

Discussion of Results

Regarding bleed capacity, the values recorded by the mixes without bentonite are similar to those shown by Krizek and Helal (1989). However, there are considerable differences from those authors' results on bentonite efficiency. While the CEMILL-S mixes (which have a B/W ratio of 4%) are perfectly stable, Krizek and Helal have recorded only small reductions of bleeding (which still remains over 30%), even with the use of large quantities of bentonite (B/W up to 13%). These surprisingly poor stabilities could possibly be due to one of two factors: a) the addition of bentonite into the water-cement grout which prevents its complete hydration, or b) the use of a dispersive additive, incompatible with the bentonite, which then flocculates and sediments. The groutability ratios proposed by Mitchell (1981) are generally supported by the grouting tests in the filters. However, near penetrability limits, mixes with the same grain size but with different stabilities and concentrations give different results. For example, the CEMILL-S mixes are more easily injected than the corresponding CEMILL-I, while all the CEMILL mixes of the same type and grain size are retained by coarser sands when made with high cement content. Mitchell therefore possibly overlooks the importance of two factors, stability and concentration, which, in borderline conditions, may be determining factors. For example, the higher the solid content, the higher the volume of the particles of large diameter that can be retained, the faster the retaining process and so the earlier the flow refusal.

The groutability limits proposed by Cambefort are generally confirmed by the tests, providing that the diameter values refer more to the D_{85} or D_{95} than to the D_{50} of the particles to be grouted. Figure 9 shows Cambefort's graph, updated with our test results. The CEMILL mixes are injectable up to the $k = 10^{-4}$ m/s limit which can, with present knowledge, be improved by simply lowering the cement content.

As regards Arenzana's relationships, only the limit $D_{10sand} \geq 0.15$ mm has been confirmed by these tests. Defining a correlation between the geometric characteristics of the mass to be grouted and those of the grout particles, the ratio between the grain diameter of the mixes and pore diameter of the soils would seem most significant. In this connection, on the basis of the tests performed, a mass would be easily groutable when the ratio between the effective diameter of the sand pores D_{30} (calculated with Kozeny) or D_{10} (measured with Hg porosimetry) and the D_{85} of the mix is respectively over 4 and 5.5, while it would not be injected when these ratios are lower than 3 and 4. In the intermediate field, the concentration and stability of the grout play a key role.

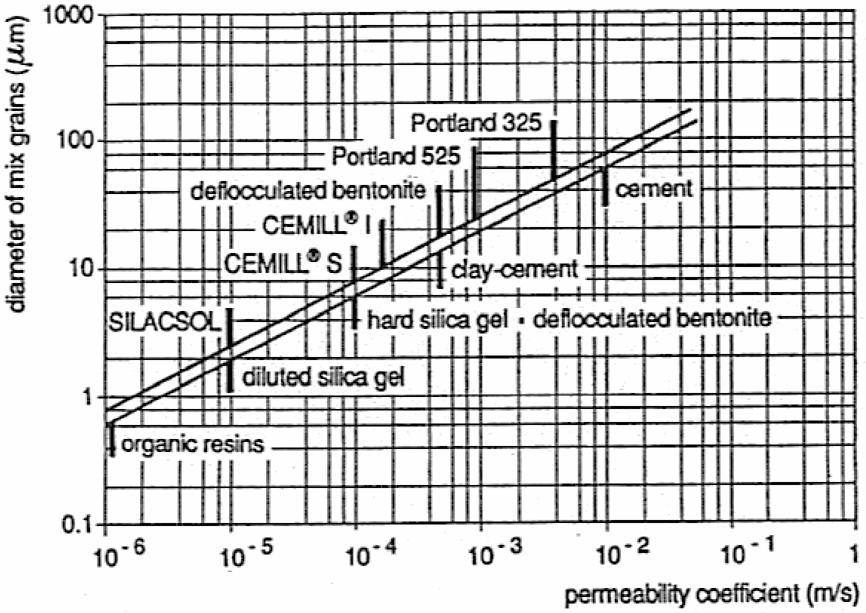

Figure 9. Penetrability limits of grouts according to Cambefort (1967) (lower) and the current Authors (upper).

Conclusions

The field production of grout mixes with microfine solid content can be made possible by the use of the CEMILL process, utilizing cement of traditional grain size. The grouts obtained may be stabilized by the addition of bentonite, which does not alter the resultant fineness of the produced mix. The rheological characteristics of the CEMILL-S mixes are close to those of the MISTRA mixes (Figure 5) (De Paoli et al. 1992). The CEMILL-I mixes have a limited stability to bleed and Newtonian rheological behaviour. The penetrability of the microcement mixes appears, however, to be governed more by the fineness of the solid component than by the rheological and stability characteristics. These do, nevertheless, retain an important influence, as does the concentration of the solid in the water, near the limit conditions of injectability. Due to their inherent stablility, the CEMILL-S mixes can provide low in situ permeabilities, even with fairly low cement contents.

The tests of penetrability in standard prefabricated filters limit the range of action of the microcement mixes to about $k = 5.10^{-5}$ m/s and therefore do not support the claim that this type of grout may completely substitute and replace certain chemical solution grouts at this time.

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