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Effect of mixing procedure on injectability of cementitious grouts

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Abstract

Injection grouts made of cement and mineral additives, such as lime and natural or artificial pozzolans, coagulate in suspension; the use of a superplasticizer is not sufficient to improve injectability when penetrability in very fine voids is required. The effect of two different mixing procedures, one with a mechanical and one with an ultrasonic mixer, has been studied. Ultrasonic mixing improves dispersion, especially when silica fume is added to the grout, and permits the use of a water content lower than that of high turbulence mixing for the achievement of the same penetrability of the grout. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Rheology; Grout; Cement; $Ca(OH)_2$; Pozzolan

Grout design involves the study of the behaviour of a suspension in the fresh and in the hardened state. The required performances of a grout at the fresh state are: high penetrability, stability of the suspension, and limited or no bleeding. In order to achieve high penetrability, the use of finely ground materials is necessary. Nevertheless, penetrability performance does not depend only on the maximum diameter of the particles contained in the grout. It is known that fine materials in suspension coagulate very easily due to interparticle interactions. The use of superplasticizers permits the development of repulsive forces due to the adsorption of the polymers on the surface of the grains. However, their action does not appear sufficient when the grout contains very fine materials such as silica fume and lime (calcium hydroxide). The particles tend to coalesce in flocs of different sizes. The penetrability capacity of such grouts is then significantly decreased. Thus, for a required penetrability performance, either the water content of the grout must increase, with detrimental effects on the stability of the suspension and the mechanical properties of the hardened grout, or the mixing procedure must be able to deflocculate the particle clusters formed in the suspension.

The action of ultrasonic waves on systems containing particles of colloidal or semicolloidal size has often been investigated. It had been demonstrated early that an ultrasonic treatment can easily disperse very fine substances such as gels or sediments [1,2]. It has further been shown that dispersivity depends on time of exposure, power, and frequency of the ultrasounds [3]. Tests with barium sulphate [3] or kaolinite and montmorillonite [4] have shown that there exists an optimal frequency for which dispersion is the best and, if the particle size increases, the frequency corresponding to the maximum dispersion is moved towards lower frequencies. The earliest reference found on the use of ultrasounds in cement and concrete technology concerns injection of prestressing ducts [5 (quoted in 6)]. More recently, the ultrasonic dispersion technique has been applied to the development of cement grouts for the repair of masonry structures [7] or for the injection of very fine soils [8].

This paper deals with the effect of the mixing procedure on the injectability performance of grouts composed of cement, lime, natural pozzolans, and silica fume of a usual particle size. The effect of each mixing procedure at different time intervals after preparation has been studied by means of the sand column test and coaxial viscometer.

1. Experimental

1.1. Materials and mixture compositions

Ordinary portland cement of the category CEM I 42.5 LA HSR (according to NBN B12-001) with low C_3A content, hydrated lime with a BET specific surface equal to 13.32 m^2/g, the natural pozzolan Rheinisch Trass, as well as silica fume in slurry, were used. The chemical analyses of the materials and the mineralogical analysis of the cement clinker are presented in Table 1. The particle size distribu-

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Table 1
Chemical composition of the materials used and minerological composition of the clinker

	Lime powder	Rheinish Trass (<80 μm)	SF	CEM I 42.5 LA HSR		Lime powder	Rheinish Trass (<80 μm)	SF	CEM I 42.5 LA HSR
L.O.I.	25.3	6.13	1.91	0.92	P_2O_5				0.18
CaO	73.2	2.16		63.22	Na_2O		1.78		
CaO free				0.66	K_2O		1.93		0.52
SiO_2	0.40	67.56	96.02	21.37					
$A1_2O_3$	0.15	16.02		3.61	C_3S				54.15
Fe_2O_3	0.10	4.56		4.13	C_2S				20.04
MgO	0.35	0.03		2.25	C_3A				2.59
SO_3	0.02	0.35		2.80	C_4AF				12.57

L.O.I. = loss on ignition; LA HSR = low alkali high sulphate resistance.

tion of cement, lime, and Rheinisch Trass after 10 min ultrasonic dispersion in alcohol are given in Fig. 1. The Rheinisch Trass was always sieved at the 80-μm sieve before use. The mixture proportions of the grouts are presented in Table 2. To increase fluidity, a sulfonated naphthalene formaldehyde based superplasticizer (SP) is preferred to a melamine-based one [9].

1.2. Grout preparation

The grouts have been prepared with two different mixing procedures. The first involves mechanical mixing with a stirrer turning at 2400 rounds per min (high turbulence mixing). All the materials were first mixed dry and then water and superplasticizer were added [10]. The second combines an ultrasonic dispersion at 28 kHz and a simple mechanical stirring (at 300 rpm). In this procedure, the materials were introduced to the water in a sequential way: the fines were mixed first and then cement was added.

1.3. Testing procedure

The penetrability performance has been tested by means of the sand-column test (AFNOR P 18-891) modified as far as the grain size of the sand is concerned. The minimal and maximal diameters of the sand used to fill the column were 1 and 2 mm, respectively. Voids with a diameter of 0.15–0.3 mm can thus be simulated [11] and all the grouts had to be injectable under a 0.8–1.0 bar pressure through this column [12]. Viscosity was measured with a Contraves Rheomat 108 E/R coaxial viscometer. Every 15 min, a small grout quantity was poured to the viscometer recipient and viscosity was thus measured. Before pouring, the mix (which was resting in a recipient) was gently remixed by hand for 15 sec, so as to be homogeneous.

2. Results and discussion

2.1. Penetrability

The first requirement for this research program was that the grouts to be further studied should be able to pass the sand column. If the compositions were not able to pass successfully through the column, the water/solids ratio and SP content were modified until a successful combination was found. The results are given in Table 3. The main differentiating factor was the silica fume (SF) content. As a matter of fact, grouts containing lime, natural pozzolan, and cement are injectable with the same water and superplasticizer content for both mixing procedures. However, the existence of small flocculates has often been observed in a grout prepared with a high turbulence mixing but has never been observed when mixing was carried out in ultrasonically. In general, these flocculates did not prevent the grout from being injectable since they were very quickly sedimenting in the recipient; nevertheless, occasionally, they obstructed the

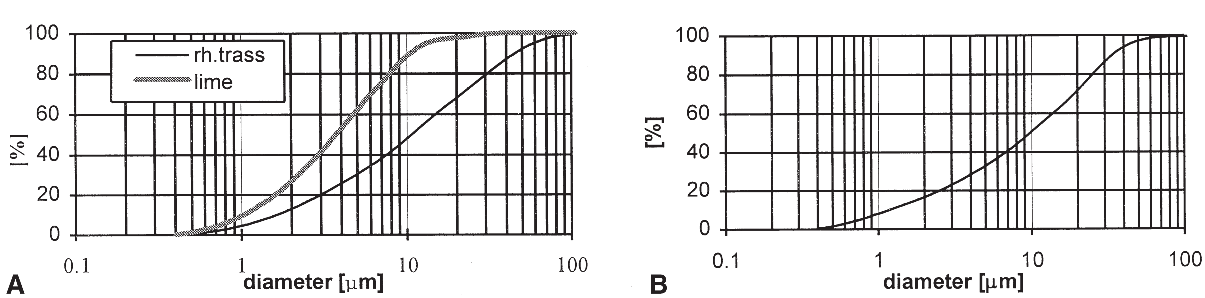

Fig. 1. Grain size distribution of the materials used: (*A*) rh.trass (non-sieved) and lime grain-size distributions, and (*B*) CEM I 42.5 LA HSR grain-size distribution.

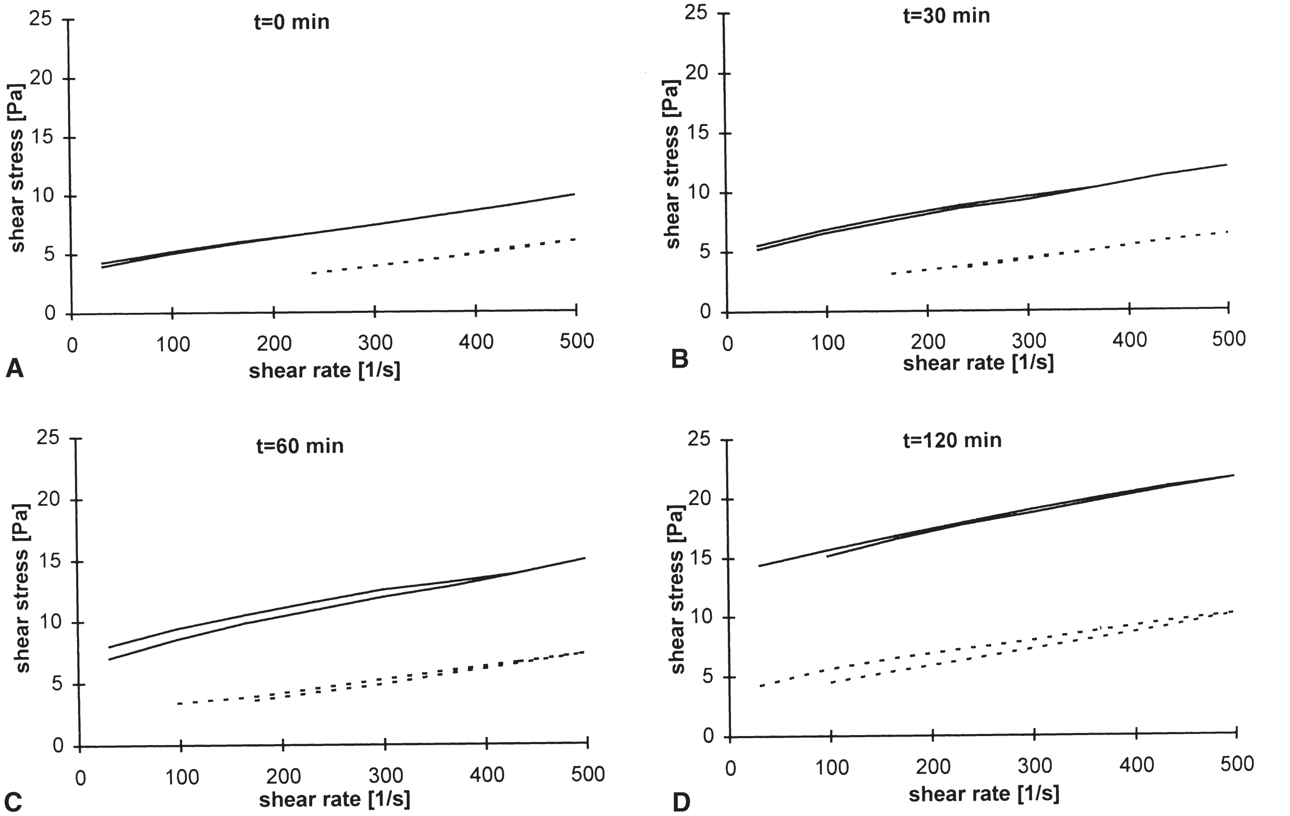

Fig. 2. Flow curves of grout 13b-0 with different mixing procedures: HT-mixing (*solid line*) and US-mixing (*dotted line*). (*A*) t = 0 min, (*B*) t = 30 min, (*C*) t = 60 min, and (*D*) t = 120 min.

exit of the grout pump (which was equipped with a net) and stopped the flow of grout through the sand column. In these cases, it was considered that failure was not attributable to the grout itself but to the mixing procedure. When silica fume was added to the grout, the high turbulence mixing procedure was not able to make the grout injectable unless a substantial change on both the water and the superplasticizer contents takes place. On the other hand, when the ultrasonic mixing procedure was applied, the grout was still perfectly injectable without any adaptation of the water and the superplasticizer contents.

Table 2
Mixture proportions of the studied grouts

Reference no. of the grout	Composition (% wt)				Lime: Rheinish Trass + SF
	Lime	Rheinish Trass	SF	Cement	
13b-0	17.5	52.5	0.0	30.0	1:3
14b-0	14.0	56.0	0.0	30.0	1:4
15b-0	11.7	58.3	0.0	30.0	1:5
13b-5	17.5	47.5	5.0	30.0	1:3
14b-5	14.0	51.0	5.0	30.0	1:4
15b-5	11.7	53.3	5.0	30.0	1:5
13b-10	17.5	42.5	10.0	30.0	1:3
14b-10	14.0	46.0	10.0	30.0	1:4
15b-10	11.7	48.3	10.0	30.0	1:5

2.2. Viscosity

In order to gain some insight on the mechanisms producing this difference, the rheological behaviour of the grouts prepared with different mixing procedures has been studied. The results related to the apparent viscosity and shear stress of grouts 13b-0 and 13b-10 are presented in Figs. 2 and 3 at four moments: immediately after mixing (0 min), 30, 60, and 120 min after preparation. The shear stress has been calculated by means of the apparent viscosity results given by the viscometer. Characteristic results of the apparent viscos-

Table 3
Penetrability conditions

Reference no. of the grout	HT mixing		US mixing	
	Water/Solids ratio	SP (% wt)	Water/Solids ratio	SP (% wt)
13b-0	0.85	1.2	0.85	1.2
14b-0	0.85	1.2	0.85	1.2
15b-0	0.85	1.2	0.85	1.2
13b-5	1.1	5.0	0.85	1.2
14b-5	1.0	5.0	0.85	1.2
15b-5	1.0	5.0	0.85	1.2
13b-10	1.1	5.0	0.85	1.2
14b-10	1.1	5.0	0.85	1.2
15b-10	1.1	5.0	0.85	1.2

HT = high turbulence; US = ultrasonic.

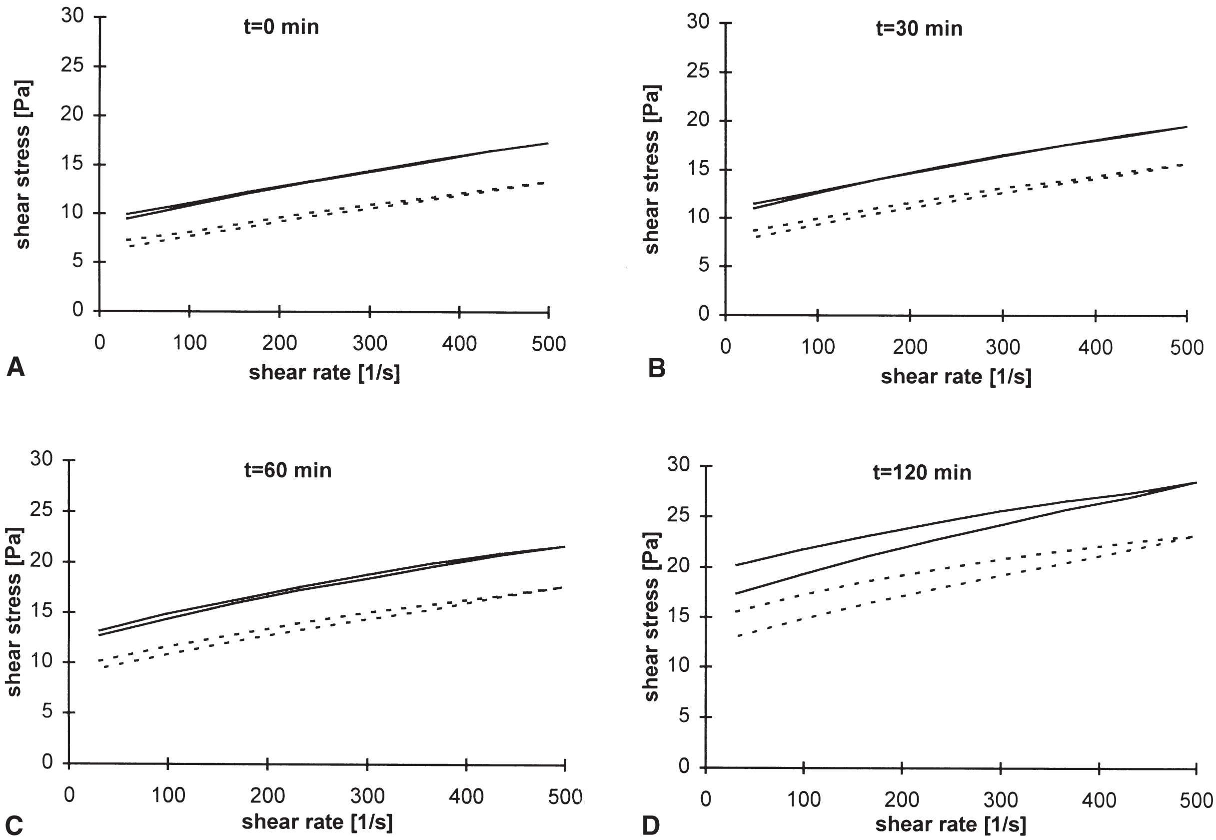

Fig. 3. Flow curves of grout 13b-10 with different mixing procedures: HT-mixing (*solid line*) and US-mixing (*dotted line*). (*A*) t = 0 min, (*B*) t = 30 min, (*C*) t = 60 min, and (*D*) t = 120 min.

ity measurements are given in Tables 4 and 5. Further, the yield stress of each composition, estimated by extrapolation of the descending branch of the shear rate-shear stress curve near the start, is presented in Table 6. The *r*-square regression coefficients are all higher than 0.98. All the rheograms, whether from blends without as well as with SF, showed a time-increasing yield stress and a thixotropic behaviour. The behaviour of the grouts can be considered to be Bingham type, at least during the period studied in this research. Despite the fact that grout 13b-0 is injectable by

Table 4
Apparent viscosity (mPa · s) of grout 13b-0 with different mixing procedures

	0 min		30 min		60 min		120 min	
Shear Rate [1/s]	HT mix	US mix	HT mix	US mix	HT mix	US mix	HT mix	US mix
29.99	142	n.m.*	173	n.m.	234	n.m.	479	140
97.14	53	n.m.	67	n.m.	88	35	161	57
164.28	36	n.m.	46	n.m.	60	25	102	39
231.42	29	15	37	16	47	21	77	31
298.57	25	14	31	15	40	19	64	26
365.71	22	13	28	14	35	17	55	24
432.85	21	13	26	13	32	16	48	22
500	20	12	24	13	30	15	43	20
432.85	21	12	26	13	32	16	48	21
365.71	22	13	28	14	36	16	54	22
298.57	25	13	32	14	42	17	63	24
231.42	29	14	38	15	50	19	77	27
164.28	36	n.m.	48	n.m.	64	22	101	32
97.14	51	n.m.	70	n.m.	97	n.m.	156	45
29.99	132	n.m.	184	n.m.	267	n.m.	446	n.m.

HT = high turbulence; US = ultrasonic.
* n.m., nonmeasurable because the torque is too low (<0.25 mN·m)

Table 5
Apparent viscosity (mPa·s) of grout 13b-10 with different mixing procedures

	0 min		30 min		60 min		120 min	
Shear rate [1/s]	HT mix	US mix	HT mix	US mix	HT mix	US mix	HT mix	US mix
29.99	332	243	383	290	438	338	673	518
97.14	114	83	131	102	153	119	224	177
164.28	75	55	85	67	99	78	141	113
231.42	58	44	66	52	76	60	105	85
298.57	48	37	55	44	63	50	86	70
365.71	42	32	48	38	55	43	73	59
432.85	38	29	43	34	48	39	63	52
500	35	27	39	31	43	35	57	46
432.85	38	29	43	34	48	38	63	50
365.71	42	31	48	37	54	42	70	56
298.57	48	35	55	42	62	48	81	64
231.42	57	42	66	50	75	57	98	77
164.28	74	53	85	64	97	74	128	100
97.14	111	79	129	96	147	111	198	152
29.99	315	219	367	266	423	313	578	435

HT = high turbulence; US = ultrasonic.

both mixing procedures, the rheograms reveal that the rheological characteristics are very different. Thus, the apparent viscosity of grouts was reduced by approximately 50–60% thanks to the ultrasonic mixing procedure.

In addition, the calculated yield stress exhibited by the same grouts was more than four times reduced due to the ultrasonic mixing. From a practical point of view, this difference suggests that a further reduction in water and superplasticizer contents is possible to achieve the same penetrability.

The presence of SF drastically modifies the rheological parameters of the grouts, but they still seem to follow the Bingham model. The yield stress is, as expected, increased but the grout still presents a thixotropic behaviour. Nevertheless, the presence of SF, especially after the first 60 min following preparation, results in a pronounced coagulation and development of a continuous structure, as is suggested by the area of the hysteresis loops of the flow curves (Fig. 3). When SF is not present, the hysteresis is much less pronounced (Fig. 2) independently of the mixing procedure used.

There exists, however, a difference between the two mixing procedures in the presence of SF as well. The decrease of the apparent viscosity was between 20–30% with the ultrasonic mixing. Further, a decrease of the calculated yield stress by approximately 25–30% has been observed. The presence of SF did not allow for the same drastic reduction in the rheological parameters as was the case with the grouts not containing SF. The action of the ultrasounds was nevertheless sufficient to make the grout 13b-10 injectable. On the other hand, grout 13b-10 prepared with the high turbulence mixing procedure is no longer injectable.

The descending branches of the experimental curves are all approximately parallel. This suggests that the mixing procedure (and the superplasticizer, of course) influence the yield stress and apparent viscosity, but the effect on the plastic viscosity is limited. The calculated plastic viscosities, with the assumption of a Bingham-type rheological behaviour as mentioned before, appear on Table 7. Plastic viscosity appears to increase with time. It also appears to slightly decrease with the ultrasonic mixing procedure. As a consequence, this factor does not seem to be able to substantially influence grout injectability, in opposition to the other rheological parameters studied.

3. Conclusions

Cementitious grout preparations by means of a high turbulence mechanical mixing procedure and an ultrasonic

Table 6
Yield stress (in Pa) of grouts with different mixing procedures

	Grout 13b-0		Grout 13b-10	
Time (min)	HT	US	HT	US
0	3.74	0.84	9.20	6.30
15	4.41	0.99	10.05	6.95
30	5.46	1.14	10.85	7.73
60	8.00	1.58	12.52	9.08
90	10.23	2.27	14.78	10.56
120	13.41	3.01	17.00	12.70

HT = high turbulence; US = ultrasonic.

Table 7
Plastic viscosity (in mPa · s) of grouts with different mixing procedures

	Grout 13b-0		Grout 13b-10	
Time (min)	HT	US	HT	US
0	12.2	10.2	16.7	14.2
15	12.3	10.1	17.4	15.5
30	13.4	10.5	17.9	16.1
60	14.2	11.4	19.1	17.4
90	15.5	12.8	21.5	18.8
120	17.2	14.2	23.4	21.2

HT = high turbulence; US = ultrasonic.

mixing procedure are compared. Their effects on some properties of grouts at the fresh state were studied. The main conclusions are as follows:

1. The high turbulence mixing procedure was found to be unable to ensure a constant penetrability of grouts composed of cement and fine materials (hydrated lime, natural pozzolan) because it is not capable of deflocculating all the formed flocs. The latter do not necessarily hinder injection since they quickly settle in the recipients; however, obstruction of pumping cannot be excluded. If the suspension contains SF, the high turbulence mixing procedure is unable to produce an injectable grout unless the water and superplasticizer contents are drastically increased.
2. The ultrasonic mixing procedure permits the production of high penetrability grouts with a limited water/solids ratio, even if SF is used. This is due to its high dispersion capacity, which permits to deflocculate even very small particle clusters.

When only normal fineness materials are to be used (such as ordinary cement, natural pozzolan sieved at the cement fineness, commercially available lime, and SF) injection grouts characterised by a very high penetrability (e.g., injectable at voids with a diameter smaller than 0.3 mm) need to be mixed by ultrasonic mixing procedure. It ensures a perfect deflocculation of the particles and therefore permits the use of limited water and superplasticizer contents.

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