

## ORDINARY PORTLAND CEMENT WITH ADVANCED FINENESS USED FOR GROUT MIXES

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*Sunt prezentate rezultatele cercetărilor de laborator privind obținerea unor cimenturi cu finețe avansată de măcinare destinate lucrărilor de consolidare prin injecție. Au fost realizate și caracterizate din punct de vedere fizico-mecanic cimenturi unitare cu diferite grade de finețe de măcinare. Cu cimenturile obținute au fost realizate fluide de injecție cu rapoarte apă/ciment variabile, utilizând aditivi superplastifianti. Fluidele de injecție au fost caracterizate din punct de vedere reologic și reotehnic.*

*The results of the laboratory investigations regarding obtaining of cements with advanced fineness designed for grouting in consolidation works are presented. Ordinary Portland cement samples with various degrees of fineness have been developed and analysed in terms of their physical and mechanical properties. The obtained cement samples were employed in preparing grout mixes with varying water/cement ratios using superplasticizers. The grout mixes have been characterised from the point of view of their rheology and rheotechnics.*

**Keywords:** Cement, Advanced fineness, Additives, Rheology, Grout mixes

### 1. Introduction

The gradual replacement in recent years of various organic chemical products, such as epoxy resins, polyurethane, acrylics, etc., with grout mixes in injection jobs has been based on a rationale which sustains and favours the present-day research in the field of ultrafine cement sorts for applications in geotechnics and construction [1–3]. A comparison of both categories of materials points out the advantages of employing grout mixes [4]:

— Grout mixes ensure the durability of the injection job due to the compatibility of the cement with the material to be grouted (for example: concrete). In contrast, for example, epoxy resins feature a relatively short shelf life thus rendering the injection process difficult to conduct and control.

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- Grout mixes preserve their structure even under varying temperature conditions thus enabling work over wide temperature intervals.
- In case of fire, grout mixes are fireproof.

Injection is now a highly efficient technology which is normally resorted to in complex applications where other solutions are either difficult or impossible to apply. However, it is also expected to soon become a technology economical enough to be employed in everyday consolidation jobs [5].

In various countries [6–9] there have been developed many ultrafine cement sorts featuring a major edge over the ordinary Portland cement as far as injection is concerned. Such advantages include: low-to-nought bleeding, high compressive strength and good injection properties, such as penetrability, stability, etc.

This paper brings information obtained by own researcher, regarding the development and characterisation of ordinary Portland cement sorts with advanced fineness. The paper presents also the characteristics of the grout mixes obtained with these cement sorts and adding various superplasticizers.

## 2. Experimentals

### 2.1. Materials

To obtain the advanced fineness ordinary Portland cement sorts, industrially-obtained clinker and gypsum were employed. Table 1 presents the modular and mineralogical composition of the industrial clinker. The content in  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  of the gypsum was 95.5%.

Table 1

Modular and mineralogical composition of the industrial clinker			
Mineralogical composition, %		Modular composition	
$\text{C}_3\text{S}$	62.32	$\text{S}_k$ <sup>*)</sup>	0.98
$\text{C}_2\text{S}$	9.63	$\text{M}_{\text{Si}}$	2.0
$\text{C}_3\text{A}$	10.47	$\text{M}_{\text{Al}}$	1.7
$\text{C}_4\text{AF}$	10.94		

\*) The lime saturation factor was calculated on the basis of the Kühl equation:

$$Sk = \frac{\%CaO}{2.8\%SiO_2 + 1.1\%Al_2O_3 + 0.7\%Fe_2O_3}$$

Note: C =  $\text{CaO}$ , S =  $\text{SiO}_2$ , A =  $\text{Al}_2\text{O}_3$ , F =  $\text{Fe}_2\text{O}_3$ ,  $\text{M}_{\text{Si}}$  = silica modulus,  $\text{M}_{\text{Al}}$  = alumina modulus.

Table 2 shows the main characteristics of the two superplasticizers employed in obtaining the grout mixes.

Table 2

### Characteristics of the superplasticizers

Superplasticizers	Main components	Density (g/cm <sup>3</sup> )
CONPLAST SP430	Naphthalene sulphonate polymer	1.20
CONPLAST SP337	Lignosulphonate and naphthalene sulphonate polymers	1.18

## 2.2. Working techniques

The grinding took place in laboratory mill,  $\Phi$  540 mm x 560 mm. The coarse grinding was carried through with a grinding media charge of approx. 144.3 kg balls. The finish grinding was carried through with an equivalent charge of double bevelled cones. The cement sorts were obtained by grinding the clinker together with the gypsum up to Blaine specific surface areas of  $7000 \text{ cm}^2/\text{g}$  and  $8000 \text{ cm}^2/\text{g}$ ,—symbolised C1 and C2, respectively.

The physical and mechanical characteristics determined on the obtained cement sorts included water demand, setting time, stability and flexural and compressive strength at 2, 7 and 28 days as per SR EN 196-1 and 3. Since a close particle size distribution was needed for the cement sorts involved in analysis, a laser particle size distribution was carried out using Malvern Mastersizer 2000E particle size analyzer.

The same ratio of superplasticizer (1%) was employed for two different water/cement ratios (0.5 and 0.6) to obtain grout mixes from the cement sorts. To characterise the grout mixes in terms of rheology and rheotechnics, determinations of fluidity and cohesion were carried out.

Fluidity was determined by timing the flow of the grout mix through the Marsh cone immediately after grout preparation (the so-called time zero) and after a subsequently 15-, 30-, 45- and 60-minute rest. Since the flowing time increases with increasing viscosity, it becomes actually an indicator of fluidity.

Cohesion was determined using a method devised by G. Lombardi [10]. The Lombardi plate cohesion meter (Fig. 1) is based on a 100 mm x 100 mm x 1.5 mm stainless steel plate used as dead-weight. The plate is immersed for a couple of seconds (2–3 sec.) into the suspension and then slowly elevated manually by spinning the spool suspending the plate. The plate is then weighed by hanging it on one side of a pair of scales with a precision of 0.1 g. Dividing the difference between the obtained weight and the dead weight by the sum of the two lateral surface areas of the plate gives the cohesion (C).

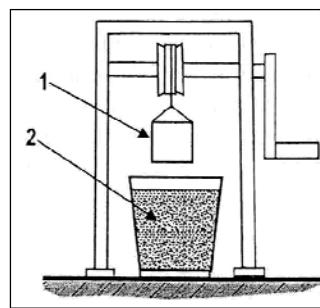


Fig. 1 Cohesion meter  
1 – Lombardi plate cohesion meter; 2 – suspension

Dividing the cohesion  $C$  by the density  $\rho$  of the suspension gives the relative cohesion  $C_r$ .

$$C_r = C/\rho \quad [\text{mm}]$$

The value  $C_r$  represents actually the thickness of the grout clinging to each side of the plate.

In order to simulate the real injection conditions, two types of Lombardi plates were employed. Horizontally grooved Lombardi plates were employed to simulate the field conditions in rocky terrain and Lombardi plates featuring both streaks and holes were employed for sandy terrain.

### 3. Results and discussion

#### 3.1. Physical and mechanical characteristics

Fig. 2 presents the particle size distribution of the advanced fineness cement sorts obtained in the laboratory against an ordinarily-ground cement sort with a  $3500 \text{ cm}^2/\text{g}$  Blaine fineness.

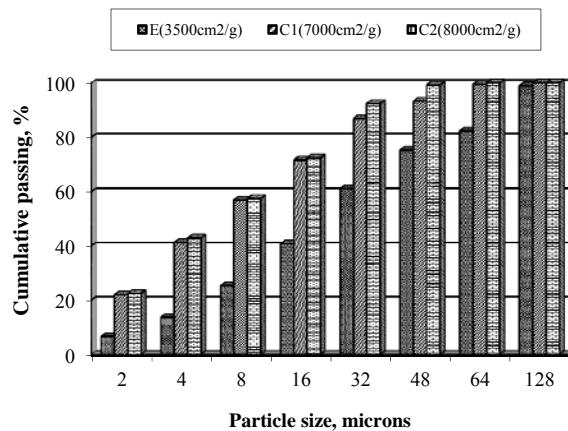


Fig. 2. Laser particle size distribution of the finely-ground cement sorts

The particle size distribution featured values consistent with the data in literature [11], namely, the particles under  $16\mu\text{m}$  reached 40.7% in the  $3500 \text{ cm}^2/\text{g}$  Blaine cement sort but went up to 71% for the C1 finely-ground cement sort with  $7000 \text{ cm}^2/\text{g}$  Blaine and up to 72.4%, respectively, for the C2 finely-ground cement sort with  $8000 \text{ cm}^2/\text{g}$  Blaine. Also consistent with the values found in literature [11], as much as 99.2% of the particles in the C2 cement sort ranged below  $48\mu\text{m}$ .

The advanced fineness and the close particle size distribution ensure the proper quality of the rheological properties required of the grout mixes employed in injection jobs [12, 13].

Table 3 presents the physical and mechanical properties of the finely-ground cement sorts, determined as per SR EN 196-1 and 3.

Table 3

**Physical and mechanical properties of the finely-ground cement sorts**

Properties		C1 cement (7000 cm <sup>2</sup> /g)	C2 cement (8000 cm <sup>2</sup> /g)
Water demand, %		28.1	30.0
Setting time	Initial, min	155	165
	Final, h-min	3-30	3-00
Stability, mm		0.5	0.5
Flexural strength, MPa	2 days	6.63	7.16
	7 days	7.24	8.10
	28 days	7.64	8.86
Compressive strength, MPa	2 days	38.9	39.8
	7 days	46.5	48.5
	28 days	54.1	57.2
Resistance class as per SR EN 197-1		52,5R	52,5R

In terms of setting time and stability, the values determined for the obtained cements sorts corresponded to the values laid down in SR EN 197-1 “Cement. Part 1: Composition, specifications and conformity criteria for cement”, namely:

- Setting time:  $\geq 75$  min
- Stability:  $\leq 10$  mm.

As expected, the advanced fineness of the cement led to an increased water demand by about 7-15% for the standard consistency as compared to an ordinarily-ground cement of 3500 cm<sup>2</sup>/g Blaine.

The mechanical strength values placed the two advanced fineness cement sorts into a superior resistance class, namely, 52,5R.

### 3.2. Rheological properties

Injection jobs require preferentially grout mixes with good fluidity and low viscosity. According to ASTM C939-10 [14], fluidity determination using the Marsh cone is especially devised for grout mixes, which feature a lower flowing time than 35 seconds. The flowing time increases with increasing viscosity; therefore, the flowing time has become a fluidity indicator. The grout mixes designed for injection jobs are recommended to feature a fluidity expressed as Marsh flowing time of 13-17 seconds right after preparation [15-17].

Fig. 3 shows the fluidity values expressed as Marsh flowing time for the grout mixes prepared from the 7000 cm<sup>2</sup>/g Blaine cement sort with water/cement ratios of 0.5 and 0.6 and different or no plasticizers.

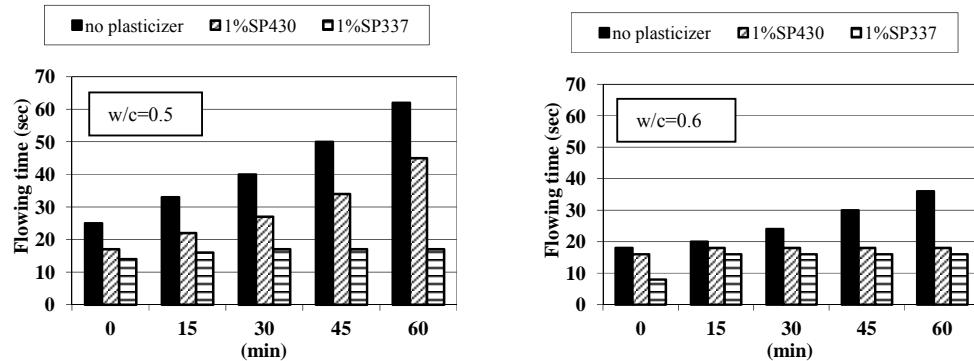


Fig. 3. Variation of the fluidity of the grout mix based on the  $7000 \text{ cm}^2/\text{g}$  Blaine cement as a function of the water/cement ratio and the superplasticizers included

The increase in the water/cement ratio from 0.5 to 0.6 determines an increase in the fluidity, with shorter flowing times through the Marsh cone.

It was found that the plasticizers had different effects on the grout mixes, namely, they determined different flowing times for the same water/cement ratio. In the case of the grout mixes based on a water/cement ratio of 0.5, the Conplast SP 430 plasticizer behaved well for the initial moment after preparation (17 sec. at time zero) but failed to help maintain fluidity over the entire duration of determination, with the fluidity of the grout decreasing gradually down to 45 seconds within 60 minutes of preparation.

In contrast to Conplast SP 430, Conplast SP 337 led to both obtaining and particularly maintaining a good fluidity (16–17 sec.) over the entire duration of determination, namely, within 60 minutes of grout preparation. In the case of the grout mixes with a water/cement ratio of 0.6, both plasticizers behaved well. The fluidity maintained a uniform value over the entire duration of determination, with the flowing time through the Marsh cone being of 14–15 sec.

Fig. 4 illustrates the variation of the fluidity of the grout mixes based on the  $8000 \text{ cm}^2/\text{g}$  Blaine cement sort.

At the w/c ratio of 0.5, the most favourable effect was that of the Conplast SP 337 plasticizer, followed by Conplast SP 430. In the case of the w/c ratio of 0.6, again it was the Conplast SP 337 plasticizer that featured the most favourable effect on fluidity. Its use led to flowing times of 13–14 sec., which are the recommended values for injection jobs.

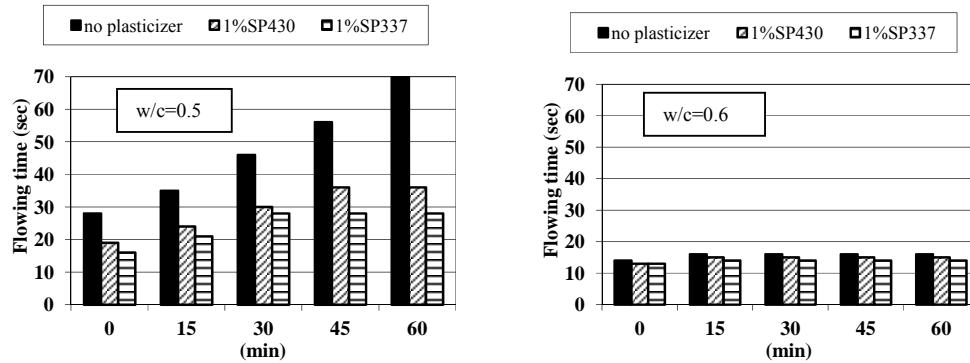


Fig. 4. Variation of the fluidity of the grout mixes based on the  $8000 \text{ cm}^2/\text{g}$  Blaine cement as a function of the water/cement ratio and the superplasticizers included

The rheological behaviour was influenced by the characteristics of the cement, particularly the fineness. The fluidity decreased with increasing fineness, which was duly correlated with the structural changes, namely, the hydration-hydrolysis processes and the stiffening and hardening processes taking place in cement over time.

The effect of the superplasticizers is due to an adsorption process at the surface of cement particles. The adsorption of the superplasticizer leads to an increased water affinity of the cement grains but also to their dispersion due to the electric charges of the plasticizer. The film of adsorbed plasticizer and the electric charges on the surface of the cement grains increase the stability of the water/cement system and, as a result, the water/cement system shows both a lower tendency to separate the water and better fluidity.

### 3.3. Rheotechnical properties

The rheotechnical properties (i.e., the cohesion) give information on the behaviour of the grout mixes during the injection work as well as on the efficiency and durability of the injection job done. An increase in the cohesion of the grout mix requires an increase in the working pressure to perform the injection. Therefore, the optimal values for the relative cohesion lie between 0.2 mm and 0.35 mm for thick grouts without any plasticizer and between 0.08 mm and 0.15 mm for grout mixes including superplasticizers [18].

Figs. 5 and 6 show the relative cohesion values for the grout mixes based on the two finely-ground cement sorts, and were obtained using the two types of Lombardi plates. Irrespective of the Lombardi plate cohesion meter employed in determining the cohesion, there was found a decrease in the relative cohesion with the increasing water/cement ratio.

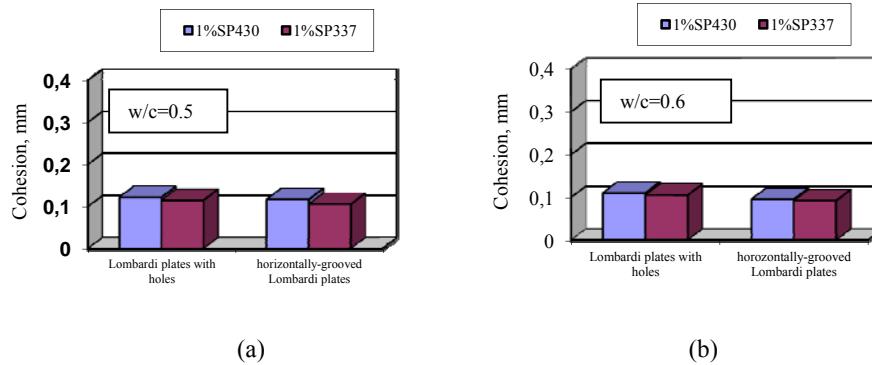


Fig. 5. Cohesion of the grout mixes based on the C1 cement sort ( $7000 \text{ cm}^2/\text{g}$ )

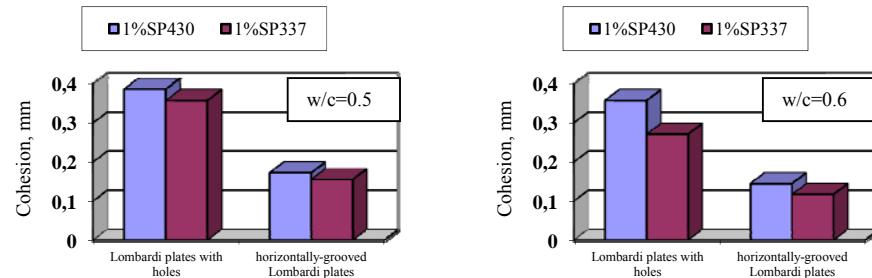


Fig. 6. Cohesion of the grout mixes based on the C2 cement sort ( $8000 \text{ cm}^2/\text{g}$ )

The fineness of the cement has a significant effect on the cohesion of the grout mixes. Thus, cohesion increases with increasing fineness. All grout mixes prepared on the basis of the C1 cement sort featured relative cohesion values of 0.09–0.12 mm, which are optimal values for injection jobs. In the case of the grout mixes based on the C2 cement sort, with a more advanced fineness, it was only for a water/cement ratio of 0.6 that optimal values for the relative cohesion were obtained when using horizontally-grooved Lombardi plates.

When discussing the effect of the type of the Lombardi plate on cohesion, it is worth noting that the cohesion was higher when Lombardi plates featuring holes were employed at water/cement ratios of both 0.5 and 0.6. Such behaviour was reiterated for both superplasticizers. Such results may be attributed to the difference in ruggedness of the plates, and, therefore, to the different capability of the plates to affect the cohesion of the paste.

On the other hand, the effect of the plasticizers on the cohesion was found to differ with the type of plate. The SP 430 plasticizer led to higher cohesion

values than the SP 337 plasticizer when using a Lombardi plate cohesion meter featuring greater ruggedness, that is, with holes.

## 6. Conclusions

- The experiments led to developing ordinary Portland cement sorts with advanced fineness ( $7000 \text{ cm}^2/\text{g}$  and  $8000 \text{ cm}^2/\text{g}$ , respectively) good to be employed in consolidation work by injection.
- The obtained cement sorts featured a close particle size distribution, with high ratios (87–93%) of particles below  $32 \mu\text{m}$ .
- In terms of mechanical properties, the advanced fineness cement sorts belongs to the resistance class 52,5R.
- The resulted cement sorts can be employed in obtaining grout mixes featuring the rheological and rheotechnical properties recommended for their use in consolidation work by injection.
  - The water/cement ratio significantly influenced the viscosity and the cohesion of the grout mixes. An increase in the water/cement ratio led to an increase in fluidity and a decrease in cohesion.
  - With their capability to disperse the cement grains, the superplasticizers led to a decrease in the flowing time through the Marsh cone (an increase in fluidity) and, at the same time, a decrease in cohesion. The best behaviour belonged to the SP337 plasticizer, based on lignosulphonate and naphthalene sulphonate polymers, which led to obtain and especially maintain a good fluidity (16–17 sec.) over the entire duration of determination, that is, up to 60 minutes from the preparation of the grout.
  - The rheological and rheotechnical behaviour of the obtained grout mixes was affected by the properties of the cement, namely, the cement fineness. The fluidity decreased with increasing fineness, while cohesion increased with increasing fineness. This was true for the lower water/cement ratios. A higher proportion of superplasticizer is necessary in this case.

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