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Development of an injectable grout for concrete repair and strengthening

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ABSTRACT

This paper deals with the coupled effect of temperature and silica fume addition on rheological, mechanical behaviour and porosity of grouts based on CEMI 42.5R, proportioned with a polycarboxylate-based high range water reducer. Preliminary tests were conducted to focus on the grout best able to fill a fibrous network since the goal of this study was to develop an optimized grout able to be injected in a mat of steel fibers for concrete strengthening.

The grout composition was developed based on criteria for fresh state and hardened state properties. For a CEMI 42.5R based grout different high range water reducer dosages (0%, 0.2%, 0.4%, 0.5%, 0.7%) and silica fume (SF) dosages (0%, 2%, 4%) were tested (as replacement of cement by mass). Rheological measurements were used to investigate the effect of polycarboxylates (PCEs) and SF dosage on grout properties, particularly its workability loss, as the mix was to be injected in a matrix of steel fibers for concrete jacketing. The workability behaviour was characterized by the rheological parameters yield stress and plastic viscosity (for different grout temperatures and resting times), as well as the procedures of mini slump cone and funnel flow time. Then, further development focused only on the best grout compositions. The cement substitution by 2% of SF exhibited the best overall behaviour and was considered as the most promising compared to the others compositions tested. Concerning the fresh state analysis, a significant workability loss was detected if grout temperature increased above 35 °C. Below this temperature the grout presented a self-levelling behaviour and a life time equal to 45 min. In the hardened state, silica fumes increased not only the grout's porosity but also the grout's compressive strength at later ages, since the pozzolanic contribution to the compressive strength does not occur until 28 d and beyond.

1. Introduction

For each application, there is a specific grout that can be adopted. Several examples can be found such as: bonded prestressed tendon grout, masonry grout, and preplaced aggregate grout, among others.

The main goal of this work was to develop an optimized grout for concrete repair and strengthening. The grout specification involves the knowledge of the flow capacity within the porous media in concrete structures and compatibility with the original materials used. Grout flow properties are affected by a large number of parameters, including binder content, type and composition, high range water reducer (HRWR) type and dosage and water/binder ratio (w/b), as well as environmental conditions (mainly moisture and temperature), water temperature, mixing time, mixer type and sequence of mixing [1,2]. A suitable grout composition, together with the flow capacity and the ability to fill the voids, will regulate the injection or placing quality.

Depending on the considered range of shear rates and grout mix-proportioning, cementitious materials may display, in steady state flow, either Newtonian, Bingham, shear thinning or shear thickening behaviour. Hence, and as a first step, the ability of a grout to perform as desired is commonly assessed using flow properties measured in the laboratory. It is clear that for injection purposes, especially in a porous media that contains fibers, the grout should behave as self-levelling, which requires a low yield stress value.

Concerning the stress generated by gravity during casting, it decreases until it becomes equal to the yield stress, at which point the flow stops [3]. Thus, in most practical situations, the knowledge of the yield stress value of a grout is very important.

Grouts and pastes are dispersions where the dispersed particles form a network responsible for the yield point. The stronger the network is, the larger the stress, so it is necessary to overcome the internal structure. If the stress applied is lower than the yield stress value it will cause an elastic deformation of the sample

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and its shape will be recovered. On the contrary, if the shear stress is higher than the yield stress (and also larger than the internal network force to resist breakage of structure), then it will result in a continuous flow.

In fact, grout or cement paste bleeding and segregation is avoided when the gravity forces, generated by the density difference in the fluid, are too small when compared to the yield stress. Besides that, there is a strong correlation between the yield stress of the cement paste and the yield stress of the associated concrete [4].

Injection grouts based on cement are colloidal suspensions in which the particle interactions may lead to the formation of various microstructures. Depending on how such structures respond to an applied shear stress or strain rate, different types of macroscopic flow behaviour can be observed. The usual ways for describing steady state flow of fresh grouts involve Bingham, Herschel–Bulkley, Ellis, Casson or Eyring rheological models [5]. With these models, it is possible to obtain grout rheological parameters, which represent a first order performance indicator for the ease of placing or injection.

Since it is also important to preserve concrete structures, the selection of grout components should also take into account the durability of the structure and the efficacy in consolidation. Partial replacement of cement by a pozzolanic material, such as silica fume (SF), generally has a desirable effect on the strength and the durability of grout, mortar and concrete [6–8]. However, two of the difficulties in using silica fume are its tendency to agglomerate and the high water demand. Because of its tendency to agglomerate, SF may persist long after the principal hydration reactions have ceased, its high agglomeration strength makes it difficult to disperse satisfactorily in the mix.

Agglomeration is also responsible for the high water demand; large amounts of mixing water are retained in the interstices within the agglomerated SF spheres. Bonen and Khayat found that SF was able to retain 40% free water after its immersion [9]. These observations have caused some authors to suggest that the primary effect of SF is generated by its physical filler properties rather than its pozzolanic properties [9–11].

Achieving predictive capabilities in a laboratory setting would be extremely useful since the injection or placing quality is crucial to obtain the best possible properties of the hardened materials. Otherwise, excessive bleeding, incomplete formwork filling, among others, can occur due to improper grout processing and injection and will affect strength and durability of the concrete structure.

Another major aspect of the rheology is the time-dependent properties of grouts, since they are fluids for which the viscosity depends not only on the shear rate applied to the sample but also on the time for which the fluid has been subjected to shearing. Thixotropic materials, such as cement-based grouts, show a shear-thinning, time dependent behaviour. As the suspension is sheared, the weak physical bonds among particles are ruptured and the network among them breaks down into separate agglomerates, which can disintegrate further into smaller flocs (structural breakdown). If the suspension is at rest, the particles will start to flocculate into agglomerates again (structural build-up), leading to a workability loss; however, a strong shearing will erase the previous effect. These structural changes are dominant and reversible but only on short observation times, which also depend on grout environmental temperature [5,12–20].

In situ grout application may occur under different environmental conditions, which may lead to different grout injection capacities, as reported by Eriksson et al. [2]. These authors noted the influence of water temperature as a factor able to influence grout properties. Considering that the application of a grout may begin early in the morning and continue throughout the day or even during several days, the variations of temperature and relative humidity become self evident, namely in the increasing of the materials' hydration reactions. Other researchers have analyzed the same influence, as is the case of Fernàndez-Altable and Casanova [21], who identified a relation between the grout rheological behaviour and the environmental temperature.

The increase of temperature and the influence of time in grout behaviour would lead to more cement particles being permanently connected, easing the workability loss. Therefore, the grout cannot flow, not even with the effort of re-agitation. Most of these phenomena are due to hydration reactions, where irreversible changes occur and thus contribute to the long term evolution of material properties (towards the solid state) [22–24].

Besides water and cement, different organic admixtures (such as superplasticizers) and mineral additives or additions may be added to yield the desired grout properties, as is the case of mix designs involving silica fume, which typically use water reducers or high range water reducers, due to the tendency of SF to agglomerate and its high water demand. The rheological behaviour of cement grout with high range water reducer depends on its type and its dosage. The addition of the HRWR causes an increase in the fluidity of the cement paste and a change in the behaviour from shear thinning to Newtonian. However, in some instances, where an excessive amount of HRWR is added, it can result in shear thickening behaviour [25,26].

It is expected that the optimum HRWR dosage is indicated by the transition from a non-Newtonian to a Newtonian behaviour. This HRWR dosage for grout could be determined using the Marsh cone test, where the optimum or saturation HRWR dosage is linked to the measured flow, beyond which there is no appreciable change in flow time. When the cement paste exhibits shear thickening, a typical non-Newtonian behaviour for over-dosage of the HRWR, the Marsh cone flow time could increase beyond the saturation dosage [26,27].

It is clear that a better understanding and classification of the basic principles that govern the fresh state properties of cement-based grouts are necessary in order to enable a composition optimization for concrete repair and strengthening and avoid the problems mentioned here. Thus, for that purpose some guidelines are presented.

Rheological measurements were used to investigate the effects of silica fume (SF) and polycarboxylates (PCEs) on cement grout properties, particularly its workability loss, as the mix will be injected in a matrix of steel fibers for concrete jacketing. The workability behaviour was characterized by the rheological parameters yield stress and plastic viscosity, as well as the procedures of mini slump cone and funnel flow time (Marsh cone test). A study was also developed which aims at contributing to better understand the flow behaviour that the grouts present under different temperatures. An attempt to find the temperature limit that isolates the thixotropy effect from hydration reactions in a grout was also made.

2. Requirements for composite design

Since the goal of this study was to develop an optimized grout able to be injected in a mat of steel fibers for concrete strengthening, it was necessary to define the requirements not only for grout design but also for the fibers.

2.1. Requirements for grout design

The main objective of this work was to evaluate a strengthening solution for reinforced concrete structures using jacketing with fiber reinforced grout. For this purpose a high performance cementitious grout reinforced with continuous and unidirectional fibers was to be developed. Thus, the qualitative requirements for this grout are the following:

- The developed grout should have a self-levelling behaviour.
- The grout should have high compressive and tensile strength properties.
- The grout should have low shrinkage.
- The grout must be easily placed/injected in the formwork that confines the fibers for concrete jacketing.
- The grout must present good durability behaviour.

This high performance cementitious grout was developed using partial replacement of cement by SF in the presence of a HRWR. Thus, the current investigation also compares the effect of various replacement levels of SF on the properties of the cement grouts.

2.2. Fiber mat requirements for grout injection optimization

The main goal of this work was to develop a high performance fiber reinforced cementitious grout with adequate properties as a jacketing material. Therefore, the required mechanical properties of the confinement grout were high compressive and tensile strengths (rather than ductility, a property that is usually obtained with the incorporation of fibers).

The mechanical properties of steel fiber reinforced concrete are influenced by various parameters, such as the type of fiber, aspect ratio (length-to-diameter ratio – l/d), the amount of fiber and the strength of the matrix [28]. The failure mode of a composite can be associated with the tensile strength of the fibers or debonding of the interface between fiber and matrix.

Thereby, continuous and unidirectional fibers (Fig. 1), set in the form of a mat, exhibited the appropriate features in order to achieve the required mechanical properties. It was expected that the use of a preplaced fiber mat (into the form) poured with a high performance grout allowed an optimization of its percentage and orientation. Besides, for continuous fibers ($l/d = \infty$), the composite should attain higher tensile strength, since the fiber embedment length is long enough to prevent fiber pullout. Thus, the expected failure is associated with the rupture of the fibers.

Fig. 1. Fiber mat.

Reference should be made to the previous efforts in this domain. For instance, in the attempt to increase significantly the mechanical properties of a steel reinforcement concrete, SIFCON (slurry infiltrated fiber concrete) [29] and SIMCON (slurry infiltrated mat concrete) [30] must be mentioned. These materials belong to the category of high performance concrete and their production process allows the incorporation of a high volume fraction of steel fibers. This process consists in preplacing the discrete fibers – SIFCON – or a fiber mat – SIMCON – into the form, followed by the infiltration of the slurry. This way, production problems such as difficulty of mixing can be avoided, allowing a higher volume fraction of fiber.

The main difference between those materials and the one that was used in the present research project is in the fiber mat, given that, in the present case, the fiber mat is made of unidirectional and continuous fibers.

3. Material characteristics

Cement: CEMI 42.5R produced in Portugal by Secil with the characteristics presented in Table 1 was employed.

High range water reducers: Modified polycarboxylates (PCEs) (Table 2).

Pozzolans: Silica fume was used due to its beneficial effects on rheology and permeability of the grout (Table 3).

The particle size distribution of the binders is represented in Fig. 2. Extensive agglomeration of the SF is indicated by its much coarser size relative to the cement.

Table 1
Chemical and physical characteristics of CEMI 42.5R (Bogue calculation).

Characteristics	CEM I 42.5R
Setting time (min)	
Start	144
End	176
Expansibility (mm)	0.60
7 d	
Flexural strength (MPa)	7.80
Compression strength (MPa)	45.90
28 d	
Flexural strength (MPa)	9.10
Compression strength (MPa)	56.30
LOI (%)	7.75
Insoluble residue (%)	1.32
SiO_2 (%)	19.56
Al_2O_3 (%)	4.23
Fe_2O_3 (%)	3.12
CaO (%)	63.30
MgO (%)	1.30
Chloride content (%)	0.02
SO_3 (%)	2.76
Free lime (%)	1.46
C_4AF (%)	9
C_3A (%)	6
C_3S (%)	68
C_2S (%)	5

Table 2
Technical and physical data provided by the manufacturer for the HRWR adopted in the tests.

Technical data	
Form	Aqueous solution of modified polycarboxylate
Density (kg/dm^3)	1.05 ± 0.02
pH-value	4.0 ± 1.0
Chloride content (%)	⩽0.1
Solid content (%)	26.0 ± 1.3

Table 3
Density and fineness using Blaine permeameter of CEMI 42.5R and Silica fume.

Sample	Density (kg/m^3)	Fineness (Blaine) (m^2/kg)
CEMI 42.5R	3140	375
Silica fume	2240	1154

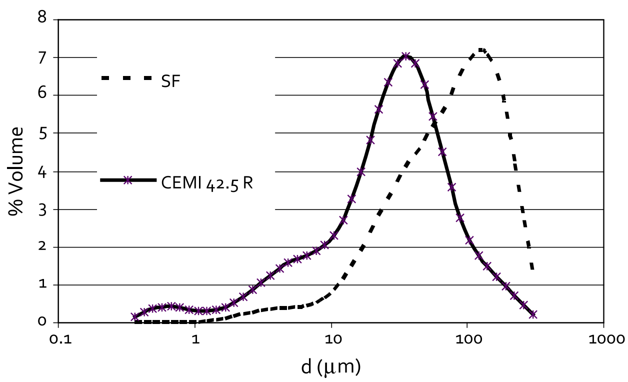

Fig. 2. Particle size distribution (in volume) of CEMI 42.5R and silica fume adopted in the tests using LASER diffraction spectrometry.

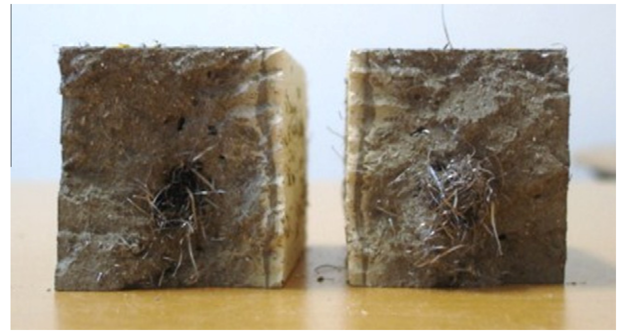

Fig. 3. Deficiencies in a cement grout (5% vol. fiber; w/c = 0.40). The grout composition was not properly optimized to minimize the fiber distribution heterogeneity.

4. Mixing and testing procedures

4.1. Preliminary tests

A set of preliminary test were carried out to assess the maximum volume fraction of fiber in the composite. The aim was to evaluate the penetrability of the matrix from a 1% up to a 5% volume fraction of fiber, without compromising the quality and the mechanical properties of the specimens. The test was made for a simple cement grout with water/cement ratios (w/c) of 0.40 and 0.28 (by weight).

In the fresh state, the workability of the mixture, the penetrability of the cementitious matrix, the quality of the specimens and the presence of voids were observed.

Segregation of the cementitious matrix was observed for a w/c ratio equal to 0.40 infiltrated in a volume of fiber up to 4% (Fig. 3). For the cementitious mixture with a w/c ratio of 0.28, it was observed that the matrix was not able to infiltrate in a volume fraction of fiber greater than 3%, leading to deficient specimens. According to Fig. 4, the presence of voids in the hardened specimens could be observed.

Taking into account the previous results and the fact that the goal was to develop a high performance cementitious grout using partial replacement of cement by SF, in the presence of HRWR, some research was made in order to identify the suitable water/binder (w/b) from a durability point of view.

Based on Mostafa's results [11], we compared the free lime contents in cement grouts with a partial replacement of Ordinary Portland cement (OPC) by SF. The pozzolan was used to replace 10%, 20% and 30% of OPC to produce blended cements and the grouts were prepared at w/b = 0.30 and 0.50 (by weight).

Fig. 5 shows the variations in the free lime contents for the mixes with time, from 1 d to 90 d.

The control samples show the highest free lime contents at all ages and the free lime contents increase continually with increasing hydration time. However, the free lime contents of blended mixes are lower than those of the control sample. This is due to the dilution effect as well as the reaction of free lime with pozzolan.

The analysis of cement grouts with w/b = 0.50 show that the concentration of free lime is higher than for those prepared with w/b = 0.30 which can be explained by the more rapid rates of hydration at the higher w/b ratio.

The pozzolanic contribution to the grout compressive strength is governed by the rate of free lime (CH%) release during hydration, which means that w/b = 0.30 leads to higher compressive strengths, as it was mentioned in [11], since it promotes the pozzolanic activity.

At this point, it was decided to keep the w/b ratio equal to 0.3 [11,31]. Concerning the fresh state, it was detected that the optimum volume fraction of fiber is less than or equal to 3%. The following step was taken to optimize the grout composition.

4.2. Rheological mix design

The main objective of this work was to evaluate a strengthening solution for reinforced concrete beams using jacketing with a fiber-reinforced grout. High strength grout is usually very viscous and not workable. As this grout was designed to be self-levelling, an optimization of the flow properties of the grout was necessary. By using rheology to optimize the flow properties, one is able to focus on each constituent of the grout composition.

4.2.1. Mixing procedures

Before preparing the grouts, the dry cement (with or without other additions) was hand mixed with a trowel to avoid the formation of granules. The mixer blade had a helicoidal shape, with a diameter (10 mm) slightly smaller than the cup diameter (11 mm) in order to allow all the grout to be mixed. The gap at the bottom, between the blade and the cup was 4 mm. Ordinary tap water was used for the preparation of the grouts. The water was allowed to flow freely until a stable water temperature was reached.

The mixing procedure was controlled to ensure that the method of mixing is representative and robust, i.e., uncontrollable variations in materials and procedures must not have a major effect. Thus, several mixing procedures were tested and the adopted mixing procedure was the following: the whole binder (cement with or without additions) is added to 90% of the water and mixed during 3 min. The remaining water is added within 30 s without stopping the mixer together with the PCE. After all materials had been added, the mixture was mixed for 3 min at 2100 rpm.

4.2.2. Marsh cone test

The Marsh cone test according to EN445 was used to optimize the HRWR dosage. The measured flow time (FT) (expressed in seconds) is the time needed to fill a 1 l container placed under the cone where the grout was introduced. The environmental conditions of the laboratory were characterized by 65% RH and a temperature of 21 ± 1 °C. The test for time of efflux was made

Fig. 4. Deficiencies in a specimen (4% vol. fiber; w/c = 0.28).

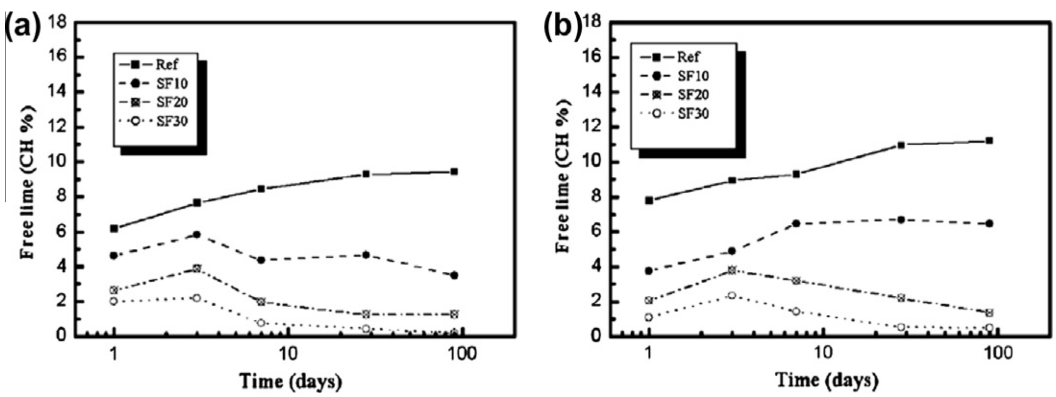

Fig. 5. Free lime contents of SF in cement grouts: (a) w/b = 0.30 and (b) w/b = 0.50, obtained from [11].

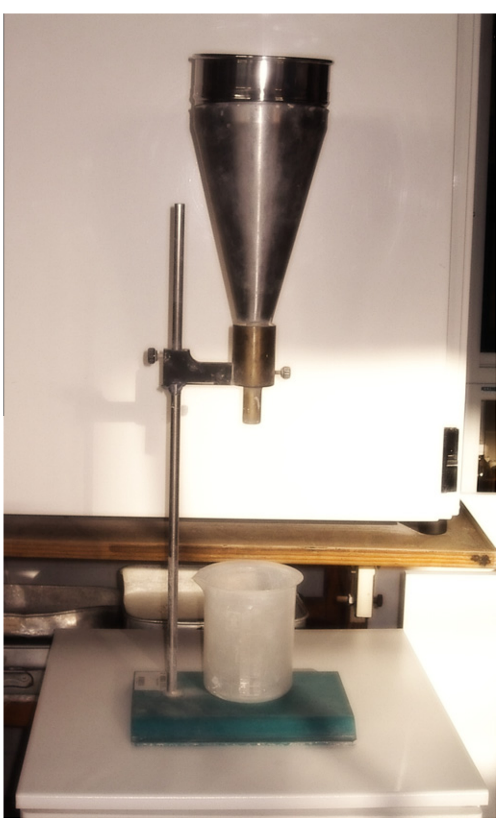

Fig. 6. Cone test device according to EN445.

immediately after grout preparation and also tested 30 min after that (Fig. 6).

4.2.3. Mini-slump test

The mini-slump test is used to determine the "workability" of fresh grout, when the yield stress value is low compared to that of concrete. In this case, a cylindrical geometry (diameter = 3 cm, height = 5 cm) was adopted and the spread was measured for the optimum grout composition, for different resting times and different environmental temperature for grout application at the laboratory and in the field. After that, an attempt to estimate the yield stress was made. The spread seems to be a more relevant parameter for estimating the material yield stress [32,33].

4.2.4. Rheological measurements

The rheological properties of the different grouts were studied using a Bohlin Gemini HRnano rotational rheometer, in a plate/plate geometry (ϕ = 40 mm), with a gap of 0.5 mm. The samples were subjected to a pre-shearing stage during 30 s at $\gamma = 300\ s^{-1}$, 8 min after binder was placed in water. After 30 s at rest, a step up test of shear rate was applied including a linear 10-min upwards from 0 to 300 s^{-1}, 60 s at maximum shear rate and an analogous step-down from 300 s^{-1} to rest (Fig. 7). The time step adopted was that

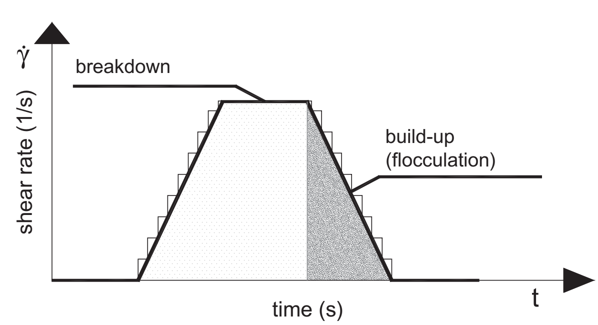

Fig. 7. Procedure for grout time independent properties and temperature effect.

necessary to obtain the steady state behaviour for each shear rate applied. For each step, the duration to reach the equilibrium state was 10 s which was revealed to be long enough to obtain steady state, but was also as short as possible to limit segregation (and of course also hydration). For each grout sample, different temperatures were applied: 5, 10, 15, 20, 25, 30, 35, 40 and 45 °C for the optimized grouts composition (for each temperature a new grout sample was used in all the tests). The procedure enabled the determination of the flocculation area, which is related to the grout workability loss before hydration.

To describe the grout rheological behaviour of cement-based grouts it was chosen to adopt the Sisko model and the modified Bingham Eq. (1) [34], since it suits better in this shear-thinning behaviour:

$$\tau = \tau_0 + \eta_p \times \dot{\gamma} + c \times \dot{\gamma}^2 \quad (1)$$

where τ is the shear stress (Pa), γ is the shear rate, τ_0 is the yield stress (Pa), η_p is the plastic viscosity (Pa s) and c is a constant.

4.2.5. Mechanical strength

Together with the previous tests, grout strength determination was made for the cement grout. In order to determine mechanical characteristics of the formulated grouts, a testing campaign was undertaken and all samples were submitted to flexural and compressive strength tests following standard NP EN 196-1.

4.2.6. Grout shrinkage

Grout shrinkage measurements were also performed since this phenomenon can lead to the formation of shrinkage cracks, which may affect the long-term performance of the grout. The method involves preparing prismatic specimens ($4 \times 4 \times 16\ cm^3$) that were removed from the molds at the age of 10 h and stored in air for 70 d at ambient temperature. Length changes of the prisms were determined weekly using a length comparator.

5. Results and discussion

5.1. Marsh cone test

The time needed for a grout sample to flow through the cone is proportional to the viscosity of the cement grout, which means that the longer the flow time, the lower the fluidity [27]. The Marsh cone value is greatly influenced by the dosage of HRWR and the optimum or saturation HRWR dosage is linked to the measured flow, beyond which there is no appreciable change in flow time. When the cement paste exhibits shear thickening behaviour, a typical non-Newtonian behaviour for over-dosage of the HRWR, the Marsh cone flow time could increase beyond the saturation dosage. The results are presented in Fig. 8.

Concerning the flow time at $t = 0$ min, several dosages of HRWR were tested: 0.2%, 0.4%, 0.5% and 0.7% by mass of cementitious material. For a grout with 0.2% of HRWR, the flow time at $t = 30$ min is too high, thus only dosages equal to or higher than 0.4% are presented in Fig. 8. As it was observed, an almost Newtonian behaviour for a HRWR = 0.5% of binder mass was detected.

Based on the desire to achieve a low initial apparent viscosity value, it was decided to use a HRWR dosage of 0.5% by mass of dry binder 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 basis of uniform HRWR dosage regardless of suspension w/b ratio was also adopted by other researchers [35].

In order to reduce the grout plastic viscosity, the dosage of silica fume was tested, also using the Marsh cone. Flow time was measured for different resting time values: 0 min, 15 min, 30 min, and 45 min. Fig. 9 presents the flow time results for different silica fume dosages.

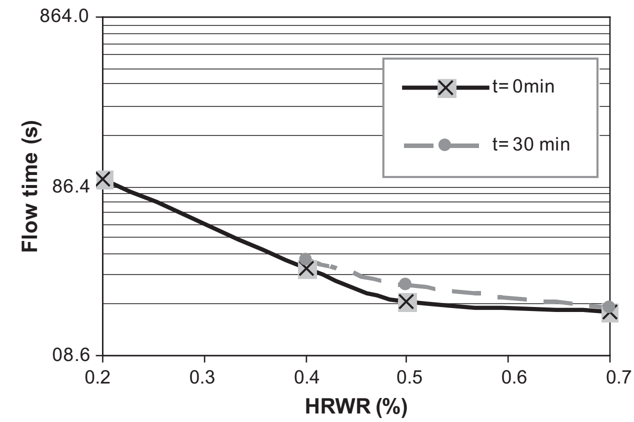

Fig. 8. Flow time for 0 min and 30 min after grout preparation for different HRWR dosage tested (% of binder mass).

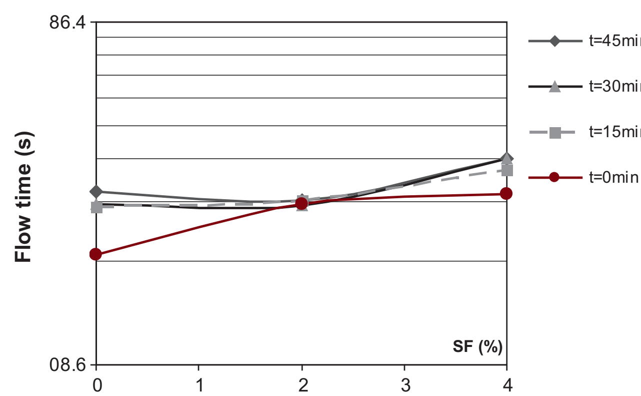

Fig. 9. Flow time for 0, 15, 30 and 45 min of resting time, after grout preparation, for different SF dosages tested (% of binder mass) and for a HRWR = 0.5%.

For a dosage higher than 2% of SF, the grout flow time tends to increase. However, that value was almost the same for different resting times tested, for a SF dosage equal to 2%. It is known that SF presents a tendency to agglomerate and increase water demand. These observations suggest that the primary effect of SF is generated by its filler properties, which explains the effect detected in flow time evolution with an increase of SF dosage.

5.2. Mechanical strength and porosity

In order to check the influence of HRWR and silica fume, a testing campaign was undertaken and all samples were submitted to flexural and compressive strength tests following standard NP EN 196-1.

Since the flexural grout behaviour will depend on fiber characteristics, in this paper only the grout compressive strength results will be presented (Figs. 10 and 11).

In the previous figures, it is possible to detect that the optimum HRWR dosage, from a strength point of view, corresponds also to the best fresh grout behaviour (HRWR = 0.5%). In fact, an optimization of the grout composition in the fresh state leads to a robust grout microstructure to beneficially influence hardened properties.

Concerning the influence of silica fume on compressive strength at early ages (namely at 7 d), it can be detected that there are no main changes if SF dosage increases from 0% to 2%. However, at 28 d there is a significant difference between the grout without

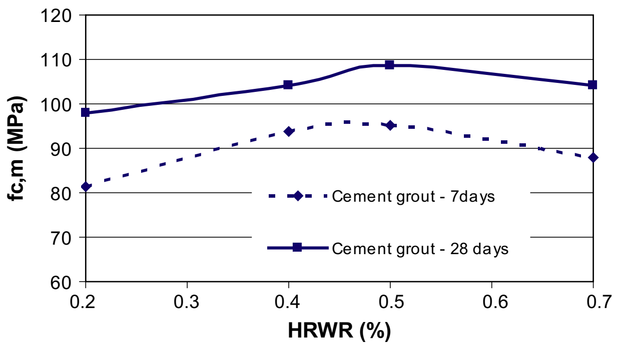

Fig. 10. Influence of HRWR dosage on grout compressive strength results at 7 d and 28 d.

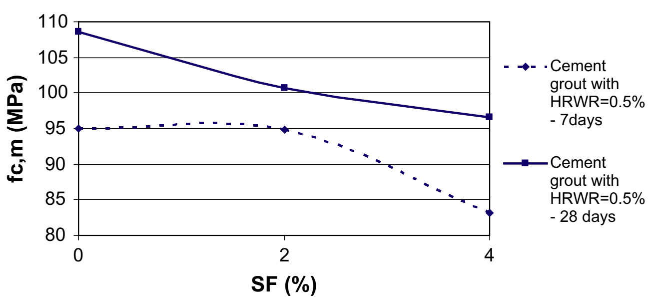

Fig. 11. Influence of SF dosage on grout compressive strength results at 7 d and 28 d (for HRWR = 0.5%).

SF and the one with 2% (cement mass substitution), where the compressive strength of the first one is higher. In addition, the strength decreased with increasing pozzolan content.

Because these mixes were made with HRWR, it was expected to obtain the filler effect of this pozzolan. However, these results indicate that at these ages SF slightly contributes as a filler material rather than a pozzolanic material which might be related to the coarser particle sizes measured for this SF in Fig. 2.

Thus, for a cement replacement between 0% and 4% by SF, it seems only to contribute to the mechanical properties just through the pozzolanic reaction, presumably at later ages.

In fact, since the grout with SF presents less clinker, it is expected that the mechanical strength does not increase as fast as for the grout with 0% of SF. The results presented in [11] also confirm this tendency. In this research [11], SF was used as pozzolan to replace 10%, 20% and 30% of OPC to produce grout. Those grouts mixes were made without HRWR, which means that the filler effects of SF were eliminated, enabling SF to contribute to the mechanical properties only through the pozzolanic reaction (Fig. 12). The same trend was observed as in our results (Fig. 13), although the present results are obtained with much lower dosages of SF than in [11].

It was expected that the addition of silica fume to the cement-based grouts decreased the porosity, increasing the packing density and thus eliminated or minimized bleeding [36]. However, the reduction detected in the strength, especially for the grout with SF = 4%, can be accounted for in terms of reduction in cement contents and increased porosity with increasing the pozzolan content at the same water/solid ratio. In fact, as the density of the SF is of 2.24 (compared with 3.14 for the OPC), a replacement by mass leads to an increase in binder volume that could partially explain the results.

In order to understand the previous results, the grouts' apparent porosity was measured using NP EN 1936:2008 (Table 4). Since the beneficial effect of SF in terms of fresh state behaviour was detected for SF = 2%, it was decided to analyze only this mix (CEMI 42.5R + 2%SF + 0.5%HRWR, with w/b = 0.30) compared to the reference grout (without SF).

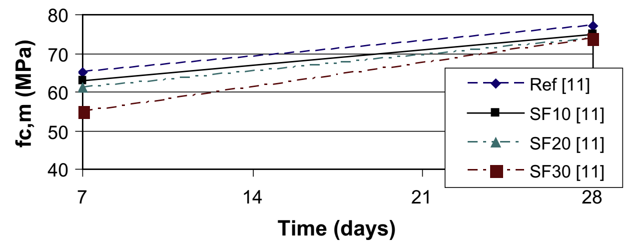

Fig. 12. Influence of SF dosage on grout compressive strength results at 7 d and 28 d, based on [11] (w/b = 0.30) (by weight).

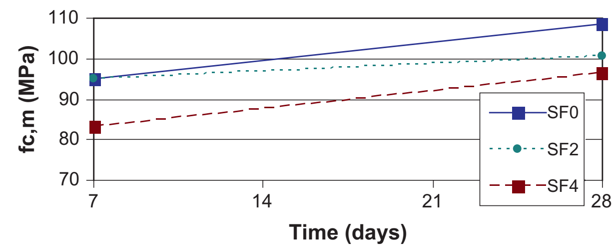

Fig. 13. Influence of SF dosage on grout compressive strength results at 7 d and 28 d (w/b = 0.30) (by weight).

Table 4
Grout apparent porosity at the age of 365 d for SF dosage equal to 0% and to 2%.

Cement grout SF (%)	Porosity (%V)
0	33.9
2	37.5

It can be observed that there is an increase of the grout porosity for an increasing SF content. This indicates that this pozzolan does not decrease the grout porosity at least for the PCE dosage adopted here, probably leading to agglomeration issues. This could also explain the mechanical strength evolution, where SF did not increase the grout compressive strength. The analysis of Fig. 2 shows that the silica fume adopted in these studies presents a particle size distribution coarser than that of the cement, which could affect the strength evolution.

The blends show higher porosity than cement grout, which is also in accordance to Mostafa's results [11]. According to this, the porosity increases with increasing pozzolan content (Table 5). This indicates that these pozzolans do not decrease the porosities by themselves.

The compressive strength was also tested at 365 d and the results for different ages (7, 28 and 365 d) were compared in terms of strength increasing due to the presence of SF (Fig. 14).

The reductions in early compressive strength and increases in total porosity of the pozzolan blends, indicate that the later improvements in compressive strength are due to the pozzolanic effect. The pozzolanic effect in this regard can be concluded as the consumption of free lime (confirmed by Fig. 5), the formation of additional C–S–H, the change in its microstructure and chemical composition (reduction in calcium/silica ratio). The reduction in the early strength can be accounted for in terms of reduction in cement contents and increased porosity with increasing the pozzolan content at the same w/b ratio (by weight). The pozzolanic contri-

Table 5
Apparent porosities of SF blended cement mixes at 90 d (based on [11]).

Mix	Porosity (%V) w/b = 0.30
Ref SF10	32.0
SF20	33.3
SF30	33.9

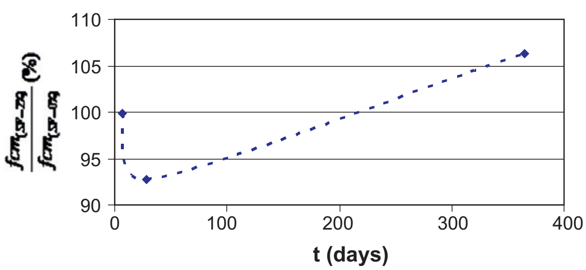

Fig. 14. Influence of SF dosage on grout compressive strength results at 7, 28 and 365 d.

bution to the compressive strength is governed by the rate of CH release during hydration and this effect does not occur until beyond 28 d.

Scanning electron microscopy (SEM) analysis was also made for hardened grouts made of CEMI 42.5R + SF = 2% + HRWR = 0.5% and for SF = 0% (w/b = 0.30) at 365 d (Fig. 15a and b and Table 6). Fig. 15a shows a sample made with SF = 0% and Fig. 15b shows a grout sample with SF = 2%.

As it can be observed in Fig. 15b, the consolidated structure indicates that the pozzolan–lime reaction has proceeded and produced more calcium silicate hydrates (C–S–H), thus combining more water and further reducing porosity when compared with the grout without SF.

The SEM is also equipped with an Energy Dispersive Spectrometer (EDS). Fig. 16a and b shows the EDS patterns of w/b = 0.30 cement grouts containing 0% and 2% of SF, respectively, and hydrated up to 365 d.

The results show that there is calcium consumption with increasing pozzolan content which is in a complete agreement with the free lime determination presented in Fig. 5, based on [11].

The previous table (Table 6) shows that SiO_2 is more available in the grout with silica fume, at the age of 365 d, which was expected due to its pozzolanic effect.

5.3. Grout shrinkage

Grout cumulative shrinkage (autogenous and drying shrinkage) was also measured for six samples of the two grout compositions:

Table 6
Chemical characterizations of grout at the age of 365 d for SF dosage equal to 0 and to 2% according to SEM/EDS results.

(%)	Cement grout (%)	
	SF = 0%	SF = 2%
SiO_2	46.6	49.4
Al_2O_3	13.5	14.2
Fe_2O_3	1.2	0.9
CaO	24.0	21.1
MgO	8.7	8.6

reference grout and the cement grout with 2% SF (w/b = 0.30) (Table 7). In Figs. 17a and b and 18 it can be observed that the shrinkage is slightly smaller for the grout with SF, which is in agreement with [37]. In fact, there is a slower evolution of the drying rate phenomena in grouts with SF, due to its small particles that fill the porous media, which explain the reduced shrinkage observed. However, the standard deviation of cumulative shrinkage for the grout with SF is higher than for the one without SF.

Taking into account all of these previous benefits of blended cement grout and in order to get the advantageous effects of SF in terms of fresh state behaviour, it was decided to select the mix with 2% of SF and to develop the characterization of this optimum grout composition from hardened and fresh state points of view. The following grout composition was then prepared: CEMI 42.5R + 2%SF + 0.5%HRWR, with w/b = 0.30 (Table 7).

5.4. Mini-slump test

The previous grout composition was then characterized from a fresh state point of view. Thus, for the cement grout with 2% of SF and 0.5% of HRWR (PCE), the spread diameter was measured for different grout resting times at two different environmental temperatures during grout application (Fig. 19). The same test was made for grout produced in the laboratory and under field conditions.

As it can be observed, the grout workability is higher for the lab environment and for both situations, a significant difference in the spread between 30 and 45 min (see Fig. 20) can be detected. This is due to workability loss.

Thus, the cement based grout with SF = 2% and HRWR = 0.5% presents an open time inferior to 45 min.

5.5. Rheological measurements

The cement based grout with SF = 2% and HRWR = 0.5% was tested at different temperatures (from 5 °C to 45 °C) and its flocculation area was calculated (Fig. 21).

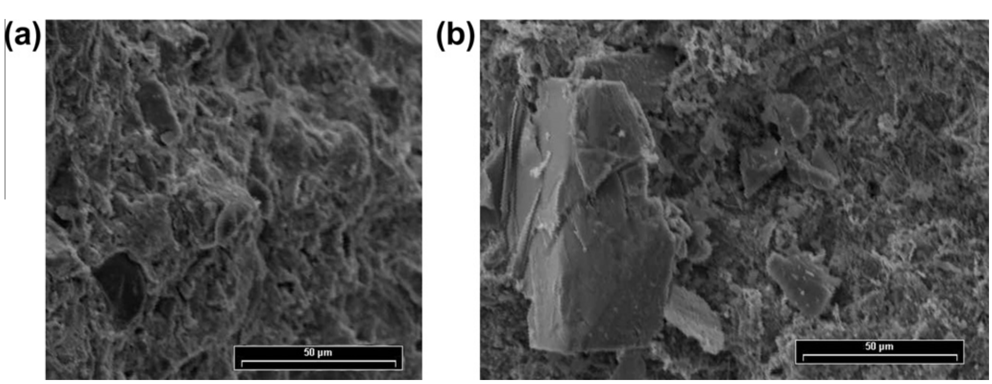

Fig. 15. Microstructure of hydrated cement grout at 365 d: (a) SF = 0% and (b) SF = 2%.

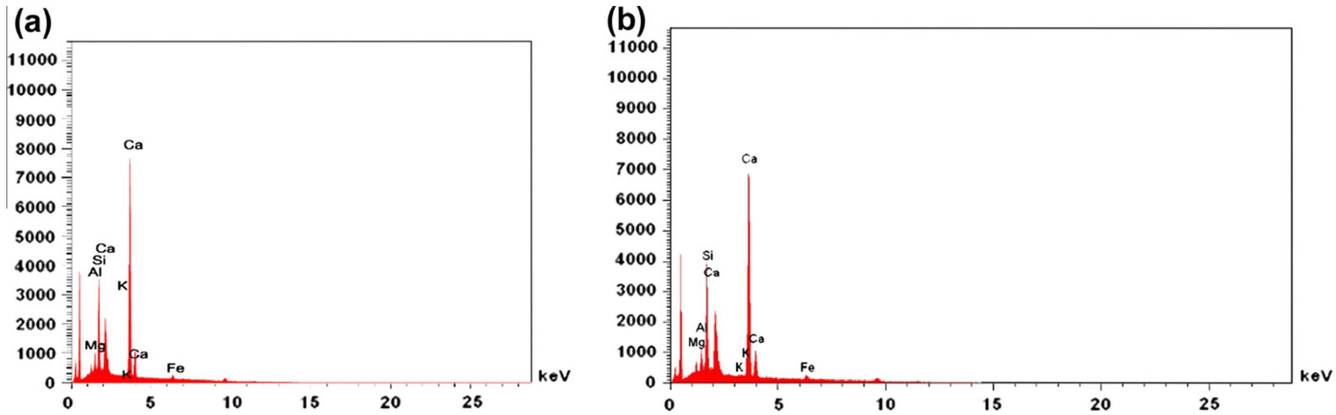

Fig. 16. Cement grout EDS spectrum at 365 d: (a) SF = 0% and (b) SF = 2%.

Table 7
Optimum grout composition tested (from a fresh state point of view).

Matrix composition		
Cement *SECIL Type I class 42.5R*	–	1536 kg/m^3
Silica fume	2% (binder wt)	31 kg/m^3
Water–binder ratio	0.30	470 kg/m^3
High range water reducer *Sika Viscocrete 3005*	0.5% (binder wt)	8 kg/m^3
Steel fiber (%V)	3%	

Like in the measurement of thixotropy in a loop test, the flocculation and de-flocculation areas also have the dimension of energy related to the volume of the sample sheared (Eq. (2)).

$$A = \mathrm{Pa} \cdot \mathrm{s}^{-1} = \frac{\mathrm{N.m}}{\mathrm{s}} \cdot \frac{1}{\mathrm{m}^3} = \frac{\mathrm{energy}}{\mathrm{volume}} \quad (2)$$

For flocculation phenomenon, the area A of the flow curve in the range 300–0 s^{-1} (step down test) indicates the energy that is required to build-up the thixotropic structure. On the other hand, de-flocculation values are related to the energy that is required to break down the thixotropic structure. This energy cannot be characterized as a material parameter in the same way as the Bingham parameters (for example), as the area A depends on the shear history of the material and degree of dispersion. However, the same experimental procedure was adopted to ensure the same grout initial conditions in order to allow a comparison of the results.

According to the previous results of grout flocculation area, it seems that there is a grout threshold temperature (T_{limit}) that separates a domain where the flocculation area is almost constant, which contrasts to another where the flocculation area starts to significantly increase. Probably, this means that in the first region ($T < 35$ °C) thixotropic effects are almost isolated from the irreversible effects (due to hydration). The same does not happen in the second region, where a temperature increase leads to faster hydration reactions. Between 40 °C and 45 °C the rheological measurements reveal an unexpected decrease. This occurs due to slippage between the matter and the plates of the rheometer, since the plates are not roughened. Because of that, only values below 40 °C are presented.

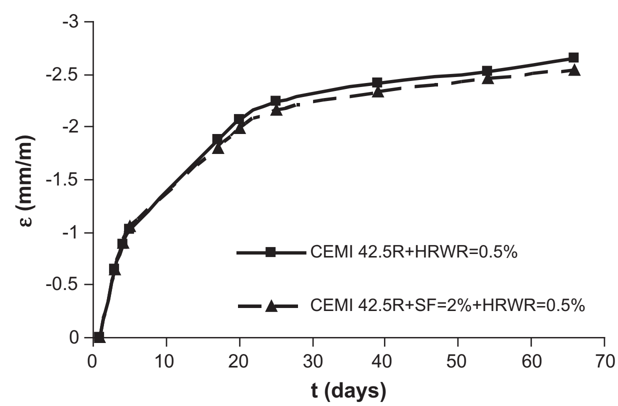

Fig. 18. Cumulative shrinkage for the reference grout and for the one with 2% of SF (w/b = 0.30) (average values).

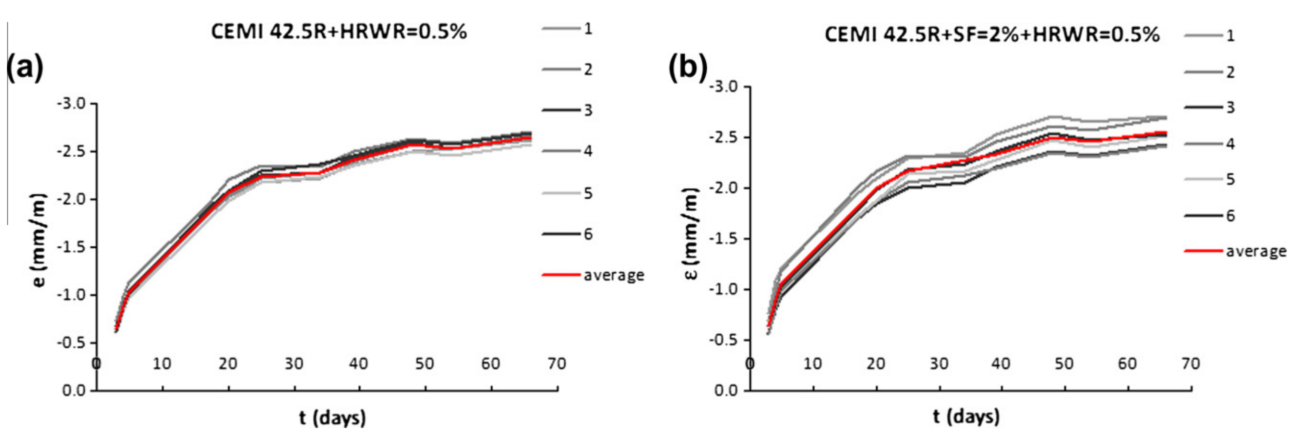

Fig. 17. Cumulative shrinkage for each samples: (a) SF = 0% and (b) SF = 2% SF (w/b = 0.30).

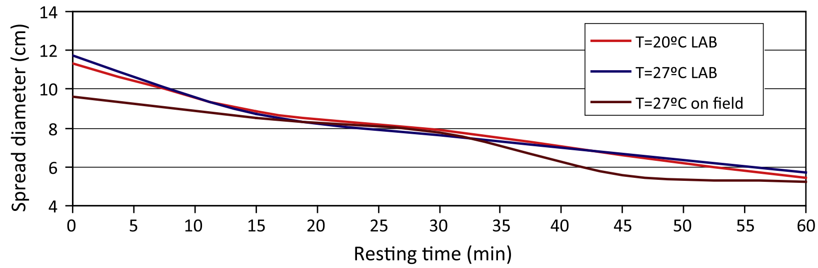

Fig. 19. Grout spread diameter evolution for different resting times.

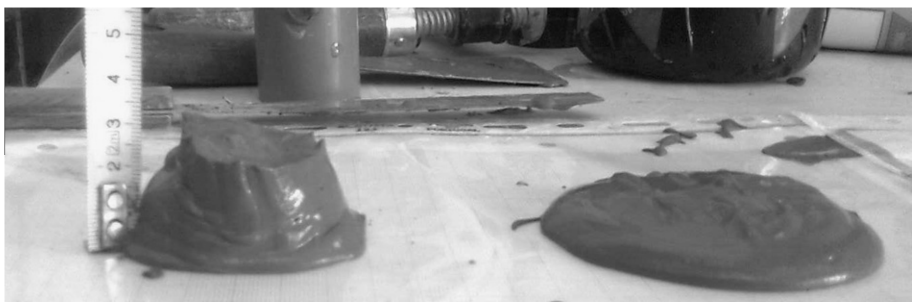

Fig. 20. left-grout spread at 45 min; right-grout spread at 30 min – for field measurements.

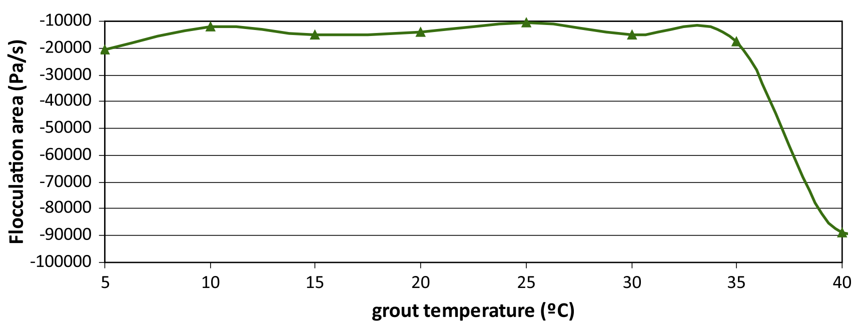

Fig. 21. Flocculation area of CEMI 42.5R + SF = 2% + HRWR = 0.5% with w/b = 0.30.

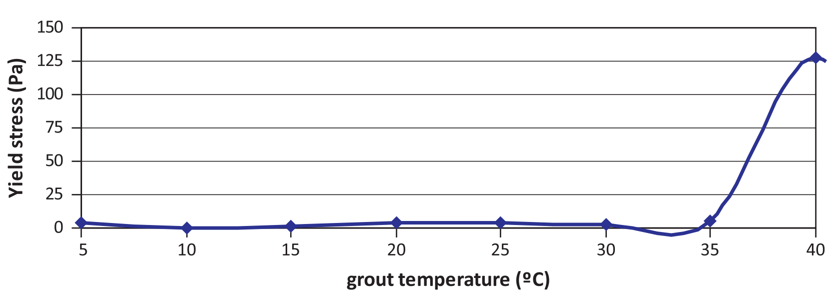

Fig. 22. Yield stress values of CEMI 42.5R + SF = 2% + HRWR = 0.5% with w/b = 0.30.

During the structural build-up, diffusion and thermal motion control the process, which leads to a long build-up time, since they lead to very small effects compared to the shear rate [34,38,39].

For the steady state analysis, the Sisko model was adopted. The modified Bingham Eq. (1) was chosen for yield stress and plastic viscosity determination. Fig. 22 presents the values of yield stress for different grout temperatures.

The previous figure also confirms that for a grout temperature higher than 35 °C an impressive workability loss can be detected. In fact, it is known that the grout presents a self-levelling behaviour for a yield stress value smaller than 50 Pa, which is in agreement with the obtained results. According to [21], the dependence of yield stress with temperature in cement pastes was found to be very specific to the combination of each type of cement and HRWR, although always uniform for each particular system. Yet, the content of tricalcium aluminate and alkali sulfates in the cement had a predominant influence on the specific trend. On the other hand, plastic viscosity always decreased with temperature until 35 °C (Fig. 23).

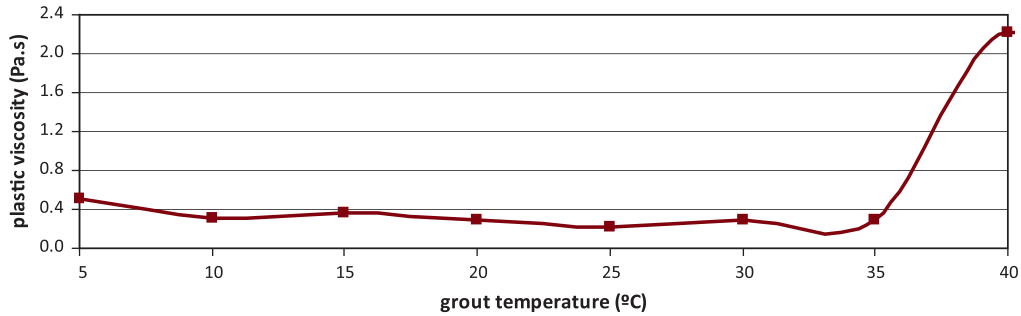

Fig. 23. Plastic viscosity values of CEMI 42.5R + SF = 2% + HRWR = 0.5% with w/b = 0.30.

6. Conclusions

The previous results of flow time and strength led us to propose the optimum grout composition (Table 7).

Concerning the fresh state behaviour and using a sophisticated rheological analysis, an impressive workability loss was detected if grout temperature increased beyond 35 °C. Below this domain, the grout presented a self-levelling behaviour and a life time inferior to 45 min.

For SF higher than 2%, the reduction detected in the early age strength can be accounted for in terms of reduction in cement contents. The pozzolanic contribution to the compressive strength does not occur until sometime after 28 d.

It was observed that there is an increase of the grout porosity for an increasing SF content at 365 d. This indicates that this pozzolan does not decrease the grout porosity, at least for the PCE dosage adopted. At this stage, the consolidated structure of the hardened grout with SF = 2% indicates that the pozzolan–lime reaction has proceeded and produced more calcium silicate hydrates (C–S–H), thus combining more water when compared with the grout without SF.

In order to produce an early age pozzolanic effect, colloidal silica could be used in future research.

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