

CONTRIBUTION TO THE DESIGN OF HYDRAULIC LIME-BASED GROUTS FOR MASONRY CONSOLIDATION

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Abstract. Grout is a concentrated suspension that can be employed for the homogenisation and consolidation of systems presenting pores, voids and cracks, such as inner core of old stone masonries. A grout should be well conceived, taking into account the optimisation of its performance; this means that simple binder and water formulations prove to be inadequate. Hence, the effect of different admixtures and pozzolans, which are traditionally used in concrete, on the fresh and hardened properties of hydraulic lime grouts, were investigated. Hydraulic lime binder plays a role of great importance in the rehabilitation of historic structures, due to its compatibility with pre-existing materials, like mortars. However, hardly any information is presently known regarding the effect of these materials on the behaviour of hydraulic lime grouts. The improvement of their properties through the use of these products is still an open field. Hence, and as a first step, the prediction of grout properties in a laboratory setting were made. The results summarised in this paper are part of a large research project and precede the fine tuning of grout composition according to the characteristics of the masonry to which such a grout is to be injected.

Keywords: hydraulic lime grout, silica fume, fly ash, superplasticiser, air entraining agent, masonry consolidation.

Introduction

Heritage or common old buildings represent the large majority of the construction types in many urban centres all over Europe. Their masonries are frequently in a bad state of conservation and present very different characteristics; some are made of a single leaf, others have multi-leaf walls. In the case of a multi-leaf wall, the section is composed by two resistant external leaves and an inner core filled by small stones, sand, mortar or other kind of unbounded material (Vintzileou 2011). The absence of cohesion among masonry elements, the existence of voids and cracks as well as the poor connection between leaves lead to masonry walls with non-monolithic behaviour. This means that the wall becomes brittle, namely under vertical and horizontal loads. In order to stabilise such walls and to prevent structural failure, grout injection is a widely used consolidation technique since the 80's (Vintzileou 2011; Miltiadou 1990).

Grout injection allows an increase in masonry compactness and creates bonds between the internal and external leaves, therefore improving the masonry's mechanical strength and monolithic behaviour. Grouts for injection should be adequately designed to achieve

the best performance from an injectability and durability point of view. This means that one of the first steps in grout optimisation for masonry injection should focus on the effects of different pozzolans and admixtures that can be used to improve their properties and to make the grout consolidation more effective.

Grouts, by definition, are mixtures of a binder and water. However, those simple mixes are unable to perform efficient consolidation, therefore requiring the use of additional products. This study concerns the use of hydraulic lime grouts with different pozzolans, such as silica fume (SF) and fly ash (FA), and admixtures, such as superplasticiser (SP) and air entraining agent (AEA). These products significantly affect fresh grout properties (rheology, stability and water retention) which are directly related with those in the hardened state (mechanical strength, porosity). FA is a by-product of coal burning from thermal electric power stations, a very fine powder with pozzolanic properties that can react with calcium hydroxide ($\text{Ca}(\text{OH})_2$) resulting from hydration of hydraulic lime. According to other researchers (Chindapasirt *et al.* 2005a, b) it is expected that the partial replacement of hydraulic lime by FA contributes to improving

grout fluidity, since the small and spherical FA particles improves contact between hydraulic lime particles by ball bearing action and reduce the friction forces. On the other hand, SF is an additive from electrometallurgy industry and can be applied as a fine filler whose particles fill the spaces made by the large and the long shape of hydraulic lime particles. Due to very fine SF particles among lime particles decrease of frictional forces between lime particles and a reduction of yield stress are expected (Lasker, Sudip 2008). Moreover, it is expected that high SF fineness increases the adsorption of water, thus allowing better water retention capabilities. However, these finer particles are the source of additional surface area resulting in an increase of contact forces among solid particles (Van-der-Waal's interactions) requiring the presence of a high range water reducer to minimize this problem, such as superplasticiser (Lasker, Sudip 2008; Artelt, Garcia 2008). According to Kadri (2009) the presence of SF also has a slight accelerating effect on the early age of strength development.

SP is a dispersant admixture whose action is based on repulsive forces; from a chemical point of view an SP is a surface active agent that acts on the binder particles by means of electrostatic charges and/or attaching long polymer chains over the particles, creating a repulsion between them (Martins, Bombard 2012). This repulsion action contributes to hold the particles far enough apart so that they cannot come together, and thus, particle flocculation is reduced or even prevented (Vickan 2005). Thus, through SP action an improvement of rheological parameters is expected, such as reduction of plastic viscosity and yield stress as well as a lower segregation. The addition of the SP causes a change in the rheological behaviour of cementitious grouts from shear thinning to quasi-Newtonian. However, it is well known that rheological behaviour of cementitious pastes with SP depends on type and dosage. AEA is a surfactant material which is a polymer molecule that has a hydrophilic polar head and a non-polar hydrophobic tail (Du, Folliard 2005). Taking into account that lime particles surfaces contain charges, the polar head group is adsorbed by the particles while the hydrophobic tail will become oriented into the air bubble present in the aqueous phase as a result of mix agitation. These small and well distributed air bubbles can act as inerts between the lime particles and the charges surrounding each air bubble causing their mutual repulsion which may improve mix fluidity (Seabra *et al.* 2009; Du, Folliard 2005; Ouyang *et al.* 2008). In addition, the adsorption of small air bubbles on the surface of the particles will prevent the sinking and consequently may improve grout stability through the reduction of segregation.

The choice of hydraulic lime (EN459-1 NHL5) as binder is a consequence of its chemical and physical properties being closer to those of the pre-existing materials in old masonries (Binda *et al.* 1997). So far admixtures and/or pozzolans have been widely used in

the case of cement based mixtures, especially in the concrete industry, to improve the strength and durability of hardened structures (Rudzirnski 1984; Demir, Baspinar 2008; Chindaprasirt *et al.* 2005b). However, little information is presently known regarding the effect of these products on the behaviour of hydraulic lime mixtures compared to common cement based mixtures (Nguyen *et al.* 2011; Yahia, Khayat 2001; Huang 2001). There are a few studies of hydraulic lime grouts involving some of these products (Papayianni *et al.* 2010; Papayianni, Pacht 2012) however, the investigation of hydraulic lime pastes is still very incipient (Bras, Henriques 2009; Bras *et al.* 2010; Baltazar *et al.* 2012). For this reason, it is important to understand and determine the rheology, stability and mechanical properties of hydraulic lime grouts containing different types of admixtures and pozzolans.

1. Scope and assumptions

This study explores the influence of two different admixtures and pozzolans on the fresh and hardened properties of hydraulic lime grouts, based on the following experimental tests: (1) rheological measurements; rheology is a powerful tool to know if a grout has a proper flowability. Good flowability is an essential criterion to allow correct grout penetration inside the porous medium (inner core of masonry) and consequently the filling of the voids and cracks. Therefore, rheological properties such as yield stress and plastic viscosity, were determined; (2) stability; the stability analysis allows quantification of phase separation phenomena. Grout stability is an essential attribute to obtain better performance from a fresh state point of view. Phase separation phenomena, such as segregation and bleeding, cause lower homogeneity in the grout during and after injection. In an unstable grout the binder particles tend to settle after being injected into masonry, which causes a stratified flow and will therefore slow down the flow; (3) water retention test; this test allows to measure the amount of water that each sample can lose. Water retention capability is another important property since it represents the ability of a grout to retain the mixing water during its injection into dry and high absorptive porous medium. The preservation of mixing water is also of great importance since it causes not only a better fresh performance but also a proper hydration of hydraulic lime; (4) air content; this test method covers determination of the air content of fresh mixed grouts. It is well known that the air content at fresh state influences not only the grout flow ability but also its strength development, thus the air entrained was investigated; (5) mechanical strength; the grout mechanical strength, particularly the compressive strength, is of great importance, since the behaviour of grout hardened has a determinant effect on the mechanical properties of the grouted masonry; (6) the open porosity is another parameter analysed; in fact water

is often present in historic buildings, which can come from different sources, and moisture transport inside the masonry is a potential cause of internal erosion of the wall. This way, it is of particular interest to have grouts with high porosity to facilitate the exit of water from masonry. Otherwise, only water vapour can be transported meaning a much slower process of losing water than when the water is able to proceed to the surface itself; (7) water vapour permeability was also analysed due to the fact that reduced water vapour permeability is a detrimental factor because it hampers the removal of water vapour which passes through masonry walls (Colleparidi 1999).

The results summarised in this paper are part of a larger research study and precede the analysis of the performance of those grouts when injected into different porous media that simulate old masonry walls (Jorne *et al.* 2012). Only then, based on the injection tests, can the composition of these grouts be finely tuned by the variation of each component's concentration according to the characteristics of masonry to which such a grout is to be injected.

2. Materials

The hydraulic lime used is a EN459-1:2010 NHL5 (2010) produced in Portugal by Secil-Martingança, which has the characteristics presented in Table 1 according to the information provided by the manufacturer. The FA used is a class F according to ASTM C216 and was collected from a Portuguese thermal power plant; a commercially available SF was used, namely undensified silica fume produced by MAPEI. The chemical composition of the lime, fly ash and silica are given in Table 2. The SP based on polycarboxylate (Glenium Sky 617) produced by BASF was used, which has the characteristics presented in Table 3. The SP used belongs to the third generation whose repulsion is a combination of coupled steric and electrostatic effects, known as electrosteric repulsion. A commercially available AEA was used (Sika Aer-5) produced by SIKA, which has the characteristics presented in Table 3.

Table 1. Natural hydraulic lime characteristics

Compression strength at 7 days (MPa)	5.5	
Fineness	90 μm	24.8%
	200 μm	2.9%
Setting time	Start	2 h 45 min
	End	6 h 37 min
Expansibility	0.79 mm	

Table 2. Chemical characterization of hydraulic lime, fly ash and silica fume

Formula	Hydraulic lime (%)	Fly ash (%)	Silica fume (%)
Al ₂ O ₃	1.43	21.86	0.15
CaO	5.54	8.23	0.2
Fe ₂ O ₃	12.54	8.97	0.03
Na ₂ O	0.96	1.78	0.05
MgO	0.78	1.74	0.3
MnO	9.75	0.03	–
SiO ₂	62.88	48.75	97
SiC	–	–	0.5
SO ₃	3.89	1.79	–
P ₂ O ₅	1.01	0.89	–
K ₂ O	0.52	1.57	0.8
TiO ₂	3.30	1.37	–
ZrO ₂	1.29	–	–
Ignition loss	19.84	1.74	2.05
Free lime	3.89	4.34	–
Blaine specific surface (cm ² /g)	5900	4010	12950
Density (g/cm ³)	2.70	2.50	2.25

Table 3. Characteristic of admixtures

Function	Water surface tension reducing	High range water reducing
Commercial name	Sika Aer-5	Glenium Sky 617
Structure of the material	Naphthalene sulphonate	Polycarboxylate
Colour	Brown	Brown
Density g/cm ³	1.04	1.05
pH	11	8
Charge	Anionic	Anionic
Chloride content %	0	<0.10
Recommended dosage range wt%	0.15–0.60	0.30–0.90

3. Experimental details

3.1. Grout compositions

The grouts were prepared with a fixed water/binder ratio of 0.5. The pozzolans (FA and SF) were blended with the hydraulic lime at replacement ratio of 10% by lime weight. The grout mixtures were prepared with and without admixtures, a SP of polycarboxylate basis (0.5% by weight of binder) was added and an AEA was used at a

Table 4. Grout mix designs

Notation	Constituents	SF (wt%)	FA (wt%)	SP (wt%)	AEA (wt%)	water/ binder (–)	Lime (g)
Mix 1	Lime	–	–	–	–		1200
Mix 2	Lime + SF	10	–	–	–		1080
Mix 3	Lime + FA	–	10	–	–		1080
Mix 4	Lime + SP	–	–	0.5	–		1200
Mix 5	Lime + SP + SF	10	–	0.5	–	0.5	1080
Mix 6	Lime + SP + FA	–	10	0.5	–		1080
Mix 7	Lime + AEA	–	–	–	0.5		1200
Mix 8	Lime + AEA + SF	10	–	–	0.5		1080
Mix 9	Lime + AEA + FA	–	10	–	0.5		1080
SF	Silica fume						
FA	Fly ash						
SP	Superplasticiser						
AEA	Air entraining agent						
W/B	Water to binder ratio						

dosage of 0.5% by weight of binder. A total of nine different grout mixtures were performed as showed in Table 4.

3.2. Mixing procedures

The hydraulic lime grouts were prepared at room temperature (20 ± 2 °C) and a relative humidity of $60 \pm 2\%$. For the preparation of grouts ordinary tap water, at a temperature of 18 ± 2 °C, was used and both pozzolans (SF or FA) and dry hydraulic lime were hand mixed to ensure a homogeneous distribution before the beginning of the mechanical mixing. The mixture procedure adopted was the following (Baltazar *et al.* 2012): the whole powder (lime + pozzolan) is added to 70% of total mix water and mixed for 10 minutes. The remaining water (with diluted SP or AEA) is added within 30 s (without stopping the mixer). After all materials had been added, the mixture was maintained for an additional 3 minutes at 800 rpm. At the end of mixing, each grout sample was passed through a sieve with 1.18 mm (no. 16 ASTM) before the experimental measurements. The delay of 10 minutes in the addition of SP makes hydraulic lime performance more effective because at early stages of hydration (first few minutes) a large amount of anhydrous phase is generated very rapidly (C_3A) and if SP is present, it becomes partly intercalated (co-precipitation) between layers of these hydrates, preventing them from exerting dispersing action (Flatt, Houst 2001).

3.3. Rheological measurements

Rheological properties were evaluated with a Bohlin Gemin HR^{nano} rotational rheometer, equipped with a plate-plate geometry (with $\varnothing = 40$ mm) and a gap of 2 mm. The grout samples were analysed 10 minutes after the mixing process had ended. In all measurements the rheological protocol adopted was the following: a pre-shearing stage of 60 s at shear rate of 1 s^{-1}

followed by 60 s at rest was applied. The pre-shearing of 60 deformation units was applied in order to ensure a similar initial state for all samples, since after mixing and depending on the time elapsed, the sample may be not exactly at the same stage and the pre-shear has the advantage of eliminate those small differences, before starting the rheological measurements. Then, the shear rate was increased from 0 to 300 s^{-1} (the maximum shear rate used). Each shear rate was applied long enough in order to ensure the attendance of the steady state, before measurements took place. The duration of each shear rate was determined through a single shear test and correspond to the time elapsed before viscosity became constant. 6 s, for shear rates up to 4 s^{-1} and 2 s for shear rates higher than 4 s^{-1} , were chosen. However, the total test time (without taking into account the pre-shear) was 2 minutes. All grout samples were analysed with a constant temperature of 20 °C, maintained by means of a temperature unit control. As mentioned above, the fresh grout properties can be used as control factors to analyse if a grout is suitable for injection, since a smaller yield value and plastic viscosity means an easier injection process and, consequently, lower pressures. Based on previous tests it is known that the rheological behaviour of fresh hydraulic lime grouts is shear-thinning, i.e. decrease in viscosity with increase in shear rate (Bras *et al.* 2010; Roussel *et al.* 2010). The results were interpreted using a rheology suspension framework, the Bingham model was used to fit the experimental data in order to determine plastic viscosity (Barnes 2000):

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

where: τ is the shear stress (Pa); τ_0 is the yield stress (Pa); η_p is the plastic viscosity (Pa.s); $\dot{\gamma}$ (s^{-1}) is the shear rate.

In order to avoid an overestimation of the yield stress consequence of poor correlation between shear thinning part of flow curve and Eqn (1); the yield stress values were obtained from the correspondent shear stress values for a small shear rate, i.e. $\dot{\gamma} = 0.5 \text{ s}^{-1}$.

3.4. Stability

Grout stability was analysed measuring density variations, a procedure developed by Van Rickstal (Rickstal 2000). The grout samples were placed in a 500 ml cup and a spherical object with a volume of 4.85 cm^3 and a mass of 34.29 g was hanging above the grout and the immersed volume was calculated for each test. The sphere undergoes buoyancy according to *Archimedes' Law*. This force varies with grout density which changes when instability leads to the deposition of particles at the bottom of the cup causing a decrease in density in the top layers.

3.5. Water retention capability

The measurement of water retention was performed in accordance with the ASTM C941-02 (2002). This test determines the time required to remove a certain amount of water from the grout sample. A depression of $5.0 \pm 0.2 \text{ kPa}$ (controlled by digital manometer) was applied to a Bruckner funnel containing 500 ml of the grout, while a graduated cylinder collected the removed water.

3.6. Air content

The evaluation of entrained air in the fresh grout was performed by means of pressure method, using a device in accordance with the EN 459-2 (2002). This device measures the air content of fresh grout based on the pressure-to-volume relationship of *Boyles-Mariotte Law*. From observation of the change in volume of the grout sample with a change in pressure, the air content of each mix can be determined. The air content test is a simple test that was performed 2 minutes after mixer process had ended.

3.7. Compressive strength

Compressive strength was evaluated by means of five samples of each mix that were poured into oiled steel moulds ($160 \times 40 \times 40 \text{ mm}$). After 5 days, the specimens were taken out of the moulds and cured in a controlled atmosphere, at $20 \pm 2 \text{ }^\circ\text{C}$ and 60% relative humidity, until the age of the compressive test. The compressive strength test was conducted at the maturity age of 7, 14 and 28 days. A pre-load of 50N was first applied before data collection commenced at a compression rate of 0.7 mm/min to failure, using a Z050 Zwick mechanical test machine with 5 kN capacity.

3.8. Open porosity

In order to analyse grout porosity the European Standard EN623-2 (1993) was adopted. The measurement method is based on the *Archimedes' principle*, and requires that the grout sample is fully saturated with water, from which

the total open pore volume is calculated. In this method, the grout sample is fully saturated, depressurising and then pressurising the sample whilst it is fully immersed in water. The grout samples were cured in a controlled atmosphere, at $20 \pm 2 \text{ }^\circ\text{C}$ and 60% of relative humidity, until the maturity age of 28 days when the porosity test was carried out.

3.9. Water vapour permeability

The water vapour permeability test was performed applying the standard EN 1015-19 (1998), which is based on the variation of weights. The protocol was the following: an aluminum capsule was part filled with deionised water (about 1 cm of water) then a piece of cotton wool was placed over the water to avoid splashing during handing of the capsule. A moisture saturated ambient condition (RH 98%) was maintained in the cup, the perimeters of which were sealed with silicone paste. After the capsule was sealed the mass of the set (capsule + sample) was measured on a precision scale of 0.001 g. The set was then placed into a climate chamber in which a stable moisture content and temperature of 40% and $23 \text{ }^\circ\text{C}$, respectively, were maintained. Under these conditions, the moisture gradient across the specimen promoted a water vapour flux. The mass of the set was recorded every 24 hours and the capsule was held in the climate chamber until a steady vapour flow was reached i.e. when the amount of water vapour through the sample per unit time reaches a constant value. Vapour diffusion coefficient was calculated at steady flow through the following expression:

$$\pi = \frac{G.E}{(S.\Delta P.35 \times 10^5)}, \quad (2)$$

where: G is the water vapour flow per unit of time at steady state (Kg/s); S is the area of the sample (75 mm diameter) (m^2); E is the sample thickness (m) and ΔP is the differential pressure of water vapour between the two sides of the sample (Pa).

3.10. SEM observations

The microstructure of different grout samples, previously cured in a controlled atmosphere, at $20 \pm 2 \text{ }^\circ\text{C}$ and 60% of relative humidity, until the maturity age of 7 days, were carried out using a ZEISS DSM 962 Scanning Electron Microscope (SEM). The samples were previously coated using a POLARON SC502 and then examined by SEM at an accelerating voltage of 5 kV.

4. Results and discussion

4.1. Rheological measurements

Plots of variation of shear stress as a function of shear rate, for the different grout mixes, were analysed. Figure 1 shows the curves obtained from the mean values of at least three repetitions of each mix (i.e. the rheological tests have been performed on three different mixes having the same composition). The coefficient of variation (CV), which is a mean variation coefficient for all the

shear rates applied on each grout composition, is also given in Figure 1. The plastic viscosity values presented in Table 5 were calculated from the fitting of Eqn (1) to the experimental data. Figure 2 shows the rheological model adjusted to the linear part of the flow curve of mix 1 and 2. The yield stress values correspond to the shear stress measured at shear rate of 0.5 s^{-1} . The authors are aware that, doing this, the values obtained may be overestimated, however they allow for comparison between different mixes.

From the results presented in Table 5 it is clear that SF has a harmful effect on grout rheological behaviour (mix 2). The addition of SF leads to an increase of specific surface, causing higher adsorption of water resulting in an increase of yield stress and plastic viscosity. According to the conclusions of Kadri and Aggoun (2009) the use of SF causes difficulties in workability of cementitious mixtures, since the cohesiveness of grout mixture improves when SF is present, which is due to an increase in the number of contact points between solid particles; thus, the presence of other admixtures such as SPs is required to solve this problem (Rao 2003). Indeed, when SP is added (mix 5) the fresh grout properties, such as yield stress and plastic viscosity, decrease compared to the grout without SP (mix 2). Note, however, that the results of mix 4 and 5 are not directly comparable, since

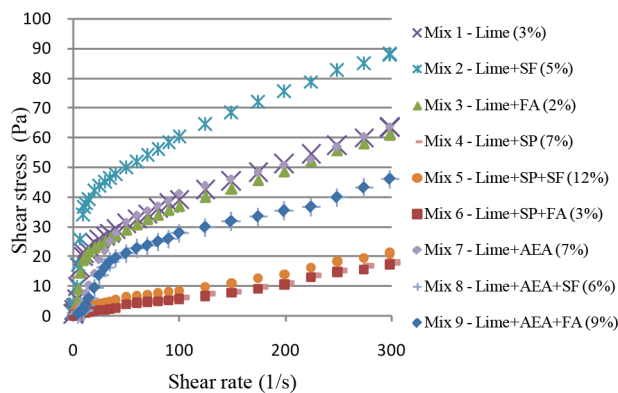


Fig. 1. Shear stress versus shear rate for different grout mixes Values in brackets give the CV

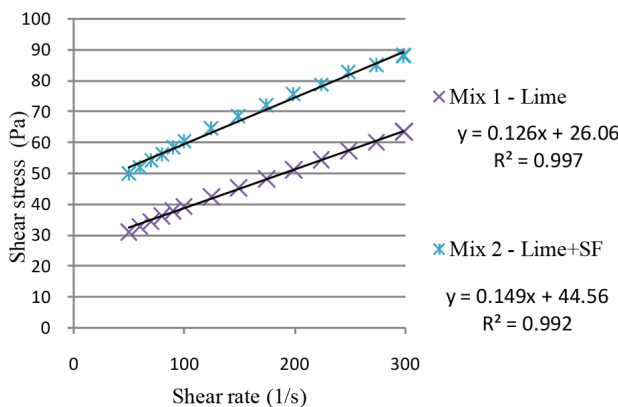


Fig. 2. Fitting the Bingham equation to the linear part of the flow curve of mix 1 and 2

the same dosage of SP has been used for all the mixes and, due to the high specific surface area of SF, the saturation dosage may be much larger for mix 5 than that for mix 4.

Hydraulic lime replacement by FA results in a slight decrease in the rheological properties. This behaviour is probably due to the replacement of irregular hydraulic lime particles by FA particles with spherical morphology (Lasker, Sudip 2008), which contributes not only to a less mono-sized suspension but also to a lubricant effect between lime particles that facilitates the beginning of grout flow (reducing the yield stress), as can be seen by comparing the yield stress values of mix 1 and mix 3. The FA does not adversely affect the rheological performance as SF does, because the FA particles are not as small as SF particles. Hence, for the same replacement percentage the FA has a lower surface area, thus resulting in a smaller adsorption of admixtures and consequently lower water consumption. This fact leads to an improvement in grout injectability through a slight reduction of yield stress and viscosity values.

From the values of rheological parameters (Table 5) it is clear that SP is the admixture that most contributes to a better grout rheological performance (as shown by mix 1 and 4, since SP imposes repulsive forces that prevent the solid particles from flocculation, resulting in a larger dispersion of the grout and a decrease in yield stress and lastic viscosity. In fact, the results show that SP incorporation is a necessity when SF is used. The grout with SP and FA (mix 6) shows the best rheological behaviour (the lowest yield stress and plastic viscosity), perhaps due to the combined effect of ball bearing action of FA particles and dispersing action of SP.

It is well known that rheological properties are affected by the air voids inside the grout microstructure. Hence, the small air bubbles, which work as a

Table 5. Rheological properties of grouts

		Yield stress (Pa)	Plastic viscosity (Pa.s)	
			Value	r
Mix 1	Lime	1.64	0.126	1.00
Mix 2	Lime + SF	2.36	0.150	0.99
Mix 3	Lime + FA	1.30	0.122	1.00
Mix 4	Lime + SP	0.50	0.061	0.99
Mix 5	Lime + SP + SF	0.70	0.064	0.99
Mix 6	Lime + SP + FA	0.11	0.059	0.99
Mix 7	Lime + AEA	1.54	0.140	1.00
Mix 8	Lime + AEA + SF	2.15	0.172	1.00
Mix 9	Lime + AEA + FA	0.79	0.133	1.00
SF	Silica fume			
FA	Fly ash			
SP	Superplasticiser			
AEA	Air entraining agent			
r	Correlation coefficient of Bingham model fitted to the linear part of the rheograms			

deformable inert in the mixture, help to disperse the hydraulic lime particles (Du, Folliard 2005). The AEA provides the production and fixation of small air bubbles on the particle surface that improves grout fluidity, owing to its ball bearing effect and without the drawback of water consumption as happens with SF or FA. That fact leads to a slight decrease in yield stress for grout with AEA (mix 7 and 9) when compared to the grouts without AEA. However, the behaviour of grouts with AEA and SF (mix 7) becomes worse. Once again this may be due to the effect of a very high specific surface area caused by SF fineness.

4.2. Stability and water retention capability

In Figures 3a, 3b and 3c the effect of each product in grout stability and water retention for the nine mixtures tested is presented. The hydraulic lime replacement by SF (mix 2), which is an ultrafine powder whose particle dimension is at least 100 times smaller than hydraulic lime particle size, results in a suspension with dominant internal friction between closely-packed particles, that cause a reduction on grout stability (as shown in Fig. 3a), probably due to the aggregated particles that tend to sink by gravity. Even so, the presence of SF together with SP (mix 5) allows a significant improvement in grout stability as shown in Figure 3b. The presence of SF decreases the amount of free water available in the mixture (water that is not adsorbed by solid particles), mitigating the phase separation effect, and therefore contributing to a higher water retention capability. On the contrary, the addition of FA, since its lower specific surface will allow a higher amount of free water, leads to lower stability and water retention capability.

As expected, the use of the SP (mix 4) also improves stability and water retention regardless of the presence of SF or FA, as can be seen by comparing the results of the Figures 3a and 3b. The influence of SP on stability and water retention is clear, resulting from several phenomena, such as: (1) the dispersing action of SP opposes sedimentation, so the sedimentation process occurs more slowly and the particles settle more homogeneously;

(2) the SP de-flocculates grout particles, allowing a higher degree of wettability and consequently reducing the amount of free water (Mikanovic, Jolicoeur 2008).

From a stability and water retention point of view the use of AEA (mix 7) leads to the worse grout performance, when compared with the plain hydraulic lime grout (mix 1). This is in contrast to other researchers who claim that air bubble adsorption on the particle's surface could prevent the particles sinking (Du, Folliard 2005). Moreover, grout performance is further worsened with the presence of SF or FA (mix 8 and 9) which is due to an increase in surface area that enhances the amount of admixture adsorbed on particle's surfaces. Therefore, a lesser amount of AEA molecules are available for air bubble formation (Du, Folliard 2005). On the other hand, it can be seen in Figure 3c that a high water retention capability along with very low stability values was obtained in mixtures with SF and FA (mix 8 and 9). This may be due to the strong segregation phenomenon, since the particles sinking to the bottom prevent the extraction of water during the water retention test (as described in the procedure 3.5).

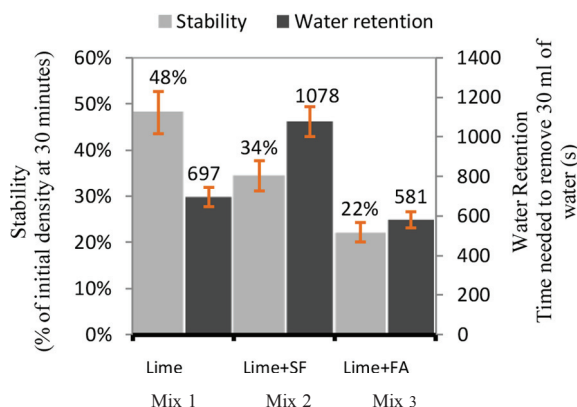


Fig. 3(a). Effect of pozzolans on stability and water retention of different grout mixes

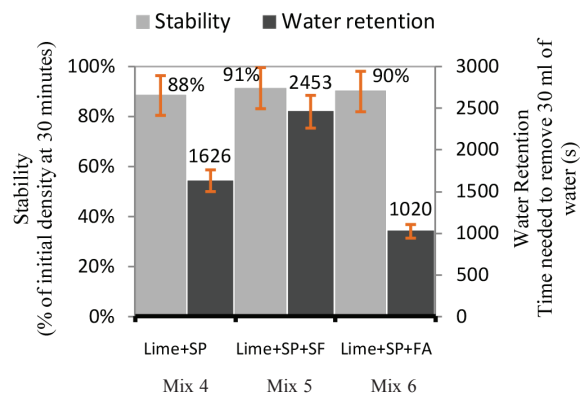


Fig. 3(b). Effect of pozzolans and SP on stability and water retention of different grout mixes

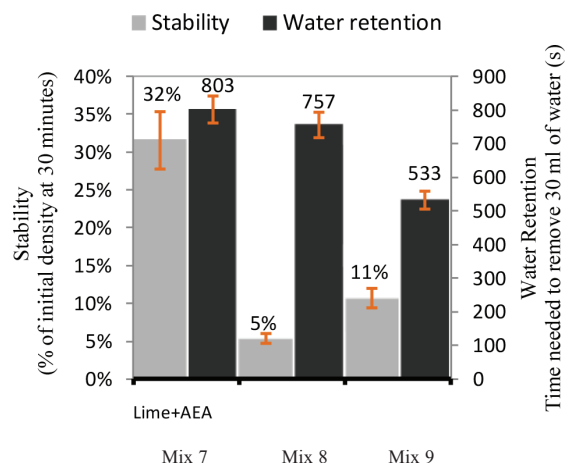


Fig. 3(c). Effect of pozzolans and AEA on stability and water retention of different grout mixes

4.3. Compressive strength and air content

The variations on compressive strength of grouts at the maturity age of 7, 14 and 28 days with different admixtures and pozzolans are shown in Figures 4a, 4b and 4c. The replacement of hydraulic lime by SF (mix 2) was found to be useful for the improvement of grout compressive strength; this results from the fact that SF leads to pozzolanic reactions, by means of reaction with $\text{Ca}(\text{OH})_2$, resulting in the formation of additional calcium silicate hydrate (C-S-H) structures (Shihada, Arafa 2010). It can be concluded from Figure 4a that SF accelerates the hydration process during the first seven days. This can be corroborated by the SEM photographs (Figs 5 and 6) where the difference is evident between the Etringite formation between the mixtures without and with SF (mix 1 and 2, respectively). The data presented in Figure 8a shows that the presence of SF leads to an increase of air incorporated in the fresh grout state. The authors attributed this behaviour to the partial replacement of hydraulic lime particles by smaller and with higher fineness SF particles, which produces an air-entraining effect. A similar effect was observed by Wang *et al.* (2010) with the incorporation of silica fume in cement mixture.

From the data presented in Figure 4a, it can be seen that FA does not significantly change compressive strength. Nevertheless, a slight decrease in grout strength can be observed when the FA is used as partial replacement of hydraulic lime. The authors are convinced that these lower compressive strengths in the FA grouts are due to the slow pozzolanic reaction of FA, since only a few parts of the FA participated in the hydration reaction during the early ages, acting only as filler (Jiang, Guan 1999). The slow hydration process caused by FA can be seen in Figure 4 by comparing the values of compressive strength between the mixtures with FA (mix 3, 6 and 9) and the plain hydraulic lime grout (mix 1). From the results of air content on fresh grout it can be seen that FA causes a similar effect as SF; although the increase in entrained air is less pronounced (Fig. 8a).

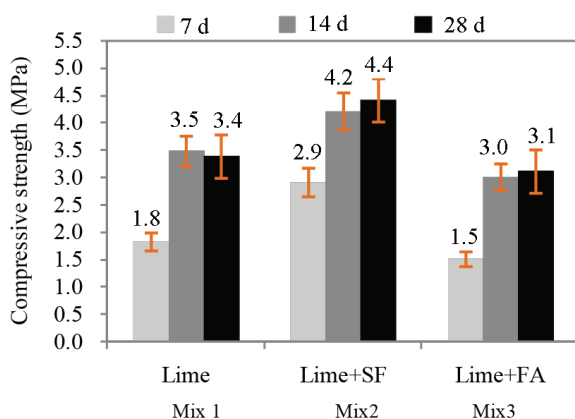


Fig. 4(a). Effect of pozzolans on the compressive strength of grouts

SP addition was found to be useful for the increment of grout compressive strength. In fact, the SP does not react by a chemical action on hydrated products; it affects the microstructure of the grout, and changes the morphology and size of lime hydration products (Demir, Baspinar 2008). The examination of the microstructure on grout fractured surface at the maturity age of 7 days shows that, instead of larger and well-defined crystals, smaller crystals are formed (Figs 5 and 7). Similar observations were noted by other authors (Yilmaz, Glasser 1989; Grabiec 1999). Cement mixtures with melamine based SP, present structures smoother and compacter when compared to cement mixtures without SP. The results obtained showed an increase in compressive strength with the presence of SP that may result from particles dispersion promoted by SP action, which simultaneously provides better lime hydration. Moreover, Fig. 8b shows that SP causes an increase in the air incorporated into the fresh grout state; however, the air entrained promoted by SP is smaller than the one caused by the AEA (Fig. 8c) as expected. Based on data presented in Figure 4 it can be noted that grout mixtures with AEA have a strength loss at the maturity age of 28 days. It is believed that strength loss may be due to expansive

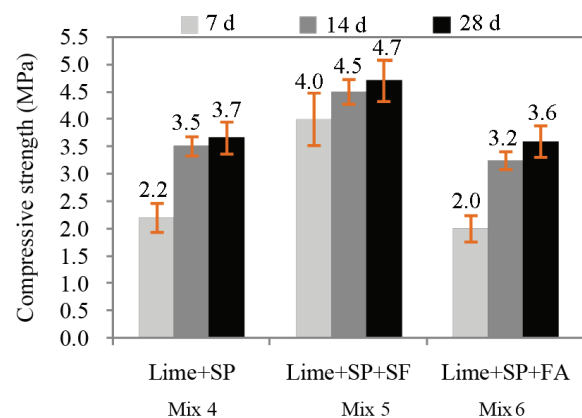


Fig. 4(b). Effect of pozzolans and SP on the compressive strength of grouts

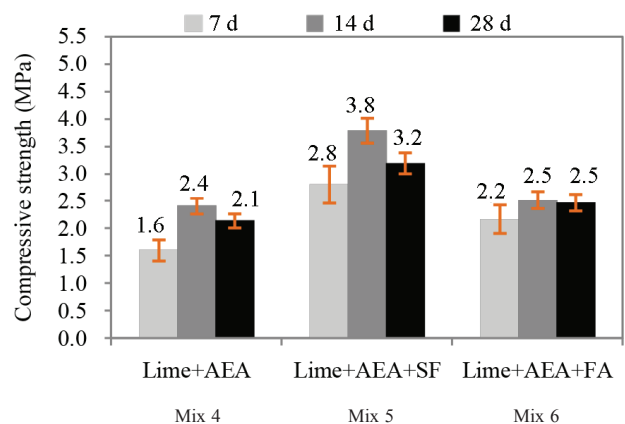


Fig. 4(c). Effect of pozzolans and AEA on the compressive strength of grouts

Ettringite formation by reaction of sulphate with anhydrous or hydrated calcium aluminates (Collepari 2003). However, this behaviour is noticeable in grout mixtures with AEA since the hardened grout structures are more brittle due to the introduction of air bubbles by means of AEA, which results in a significant decrease in compressive strength.

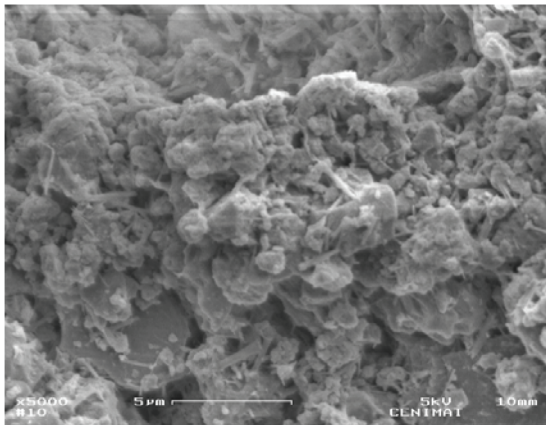


Fig. 5. SEM image of 7-days hardened grout without additives (mix 1) at 5000x

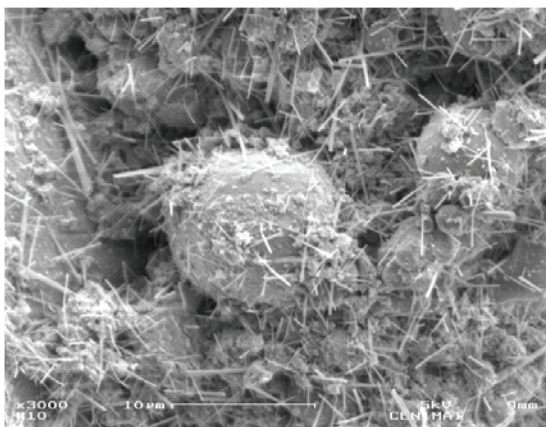


Fig. 6. SEM image of 7-days hardened grout with SF (mix 2) at 3000x

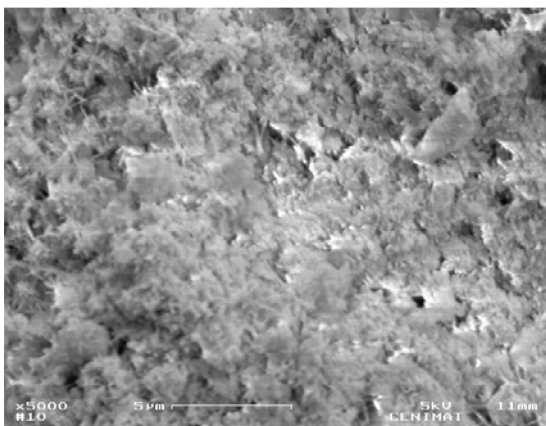


Fig. 7. SEM image of 7-days hardened grout with SP (mix 4) at 5000x

4.4. Water vapour permeability and porosity

According to the results presented in Figure 9 there is hardly any variation in grout porosity when the different admixtures and/or pozzolans were used. However, changes can be observed in water vapour permeability for the different grout compositions. SF and FA

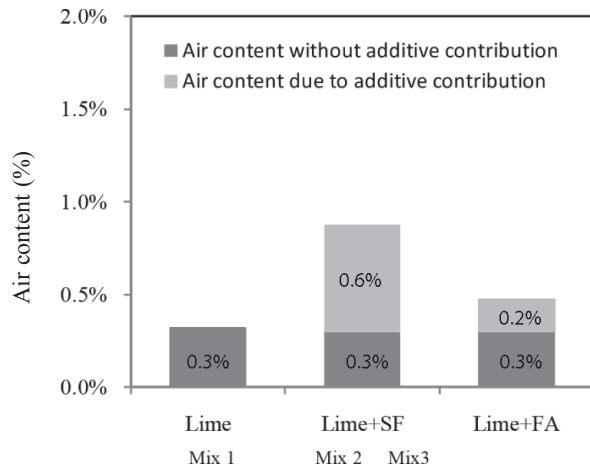


Fig. 8(a). Effect of pozzolans on air content of fresh grouts

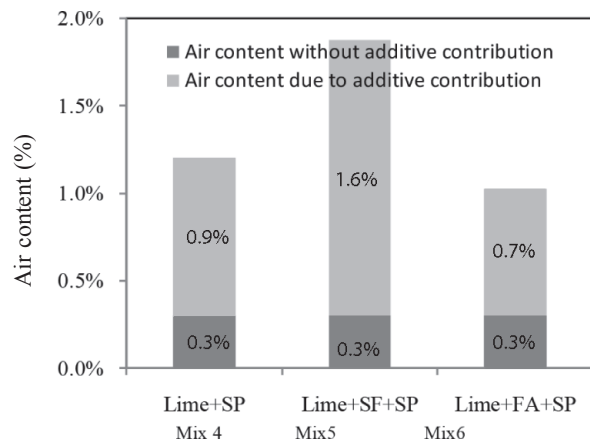


Fig. 8(b). Effect of pozzolans and SP on air content of fresh grouts

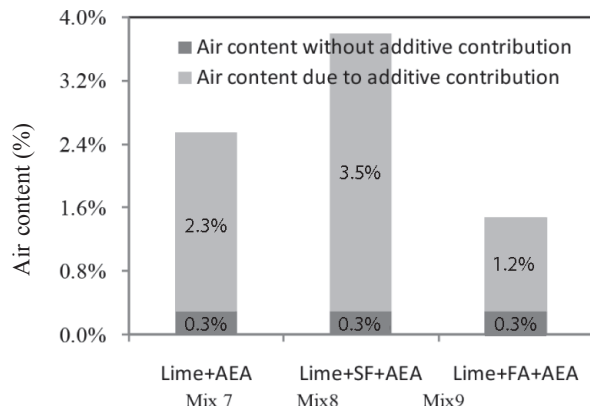


Fig. 8(c). Effect of pozzolans and AEA on air content of fresh grouts

(Fig. 9a – mix 2 and 3) cause reduction of water vapour permeability as a consequence of their contribution to lime hydration and, consequently, to the formation of hydration products, which in turn leads to changes in the grout structure at the hardened state, as previously mentioned. On the other hand, AEA (mix 7) causes a slight increase in permeability, which is expected since this admixture contributes to the dispersion of the mixture, leading to higher pores, despite the fact that the overall porosity remains unchanged. This effect is due to the

tortuosity of the pore system. The tortuosity has nothing to do with the open porosity but entirely depends on the shape of pores and in an indirect way on the connections of the pore system (Hall, Hoff 2002). This means that for the same open porosity, we can have a very complex network of pores with very narrow throats and greater length of the path described by the pore space, which hinder the water vapour transport. In the opposite situation, such as with a very regular pore structure (low tortuosity) it facilitates passage of water vapour through the pore system, which provides grout with higher water vapour permeability. So, it can be noted that open porosity has little influence on water vapour permeability, since the same porosity provides different values of water vapour permeability.

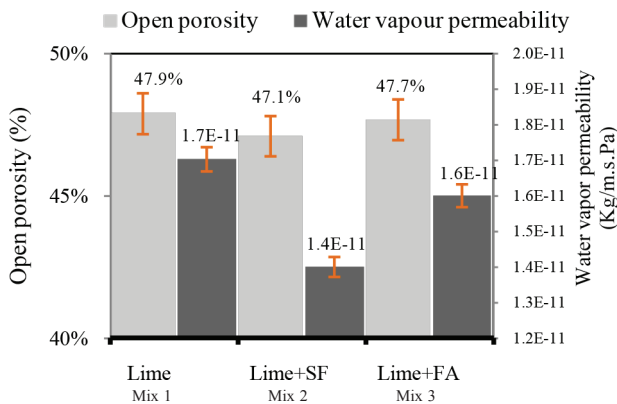


Fig. 9(a). Effect of pozzolans on water vapour permeability of grout

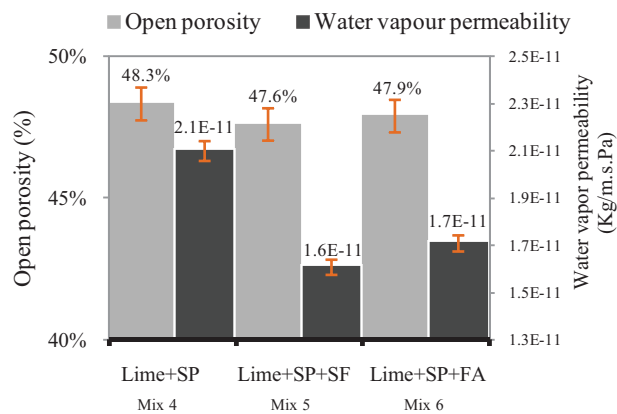


Fig. 9(b). Effect of pozzolans and SP on water vapour permeability of grout

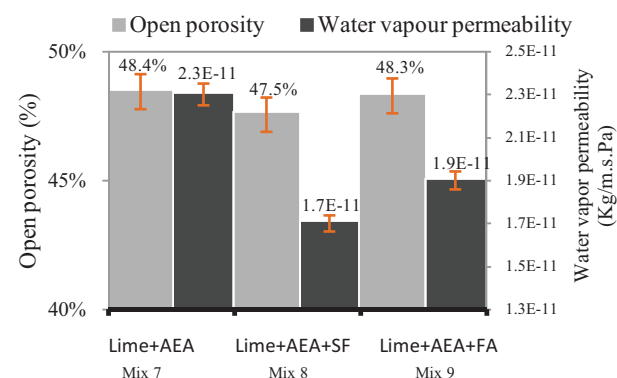


Fig. 9(c). Effect of pozzolans and AEA on water vapour permeability of grout

Conclusions

From the results presented, the following conclusions can be drawn on the influence of different admixtures and pozzolans on some relevant properties of hydraulic lime grouts, from the masonry consolidation point of view:

1. Silica fume has a harmful effect on grout rheological behaviour, causing an increase of yield value and plastic viscosity. However, the replacement of hydraulic lime by silica fume (mix 2) was found to be useful for the improvement of grout compressive strength.
2. The hydraulic lime replacement by fly ash results in a slight decrease in yield stress. Nevertheless, the presence of fly ash leads to a decrease in grout compressive strength.
3. None of the admixtures or pozzolans analysed produced a variation in grout open porosity. However, a slight increase in water vapour permeability in the presence of superplasticiser and air entraining agent was observed.
4. The results indicate that superplasticiser is the admixture that most contributes to a better grout performance, from the rheological, stability and water retention point of view.
5. Superplasticiser addition was found to be useful for the increment of grout compressive strength.
6. The presence of silica fume together with superplasticiser allows a significant improvement in the grout stability, as shown by mix 5.
7. The grouts with fly ash and superplasticiser show the best rheological behaviour i.e. the lowest yield stress and plastic viscosity.
8. Air entraining agent is by far the worst admixture, since it leads to the worst grout performance, even when compared with the plain hydraulic lime grout.

It is clear that, from a comprehensive point of view considering fresh and hardened-state results, products like superplasticiser are inevitable when designing a grout for injection purposes and its presence seems to be particularly required when other powder admixtures are used, such as silica fume. The authors recognise that these

results should not be generalized, since the influence of the analysed admixtures will depend on other parameters that have not been considered in this study, such as the temperature conditions during mixing and grouting. In any case, this investigation demonstrated the potential of some products in the improvement of fresh and hardened properties of hydraulic lime grouts and precedes the grout composition optimisation and also the analysis of the performance of those grouts when injected into different porous media that simulate old masonry walls (Jorne et al. 2012). It is believed that only in this way can long-term performance of grouts be envisaged and properly evaluated.

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