Structural Analysis of Historical Constructions – Jerzy Jasieńko (ed)
© 2012 DWE, Wrocław, Poland, ISSN 0860-2395, ISBN 978-83-7125-216-7

HYDRAULIC LIME GROUTS FOR MASONRY INJECTION – EFFECTS OF ADMIXTURES ON THE FRESH GROUT PROPERTIES

Luis G. Baltazar[1], Fernando M.A. Henriques[2], Fernando Jorne[3]

ABSTRACT

The old stone masonries especially in the case of multi-leafs walls often present low compactness and very few links between the internal and external leaves. The injections of grouts in the core of the multi-leafs masonries is a current technique used for structural consolidation. The properties of fresh grouts govern their ability to flow in the porous media and to fill the internal voids. To ensure an adequate flow of the grout inside the masonry it is essential to assure good fresh properties, such as high stability and good rheological behaviour. This research presents the effects of different admixtures, such as silica fume, fly ashes, superplasticizer and air-entraining agent on the rheological behaviour, water retention and stability of hydraulic lime grouts. This study has particular relevance due to lack of information about the use of admixtures in hydraulic lime grouts compared to the amount of information that exists in the case of Portland cement.

The results obtained demonstrated that superplasticizer is the admixture with the most expressive effect in improving the fresh grout properties. Silica fume is the second most influential admixture; however it has an undesirable effect, since the overall rheological properties become worse in the presence of silica fume. On the other hand, the air entraining agent and the fly ashes do not show a significant role to change the fresh grout properties. The research shows that there are relevant differences in the fresh behaviour of hydraulic lime grouts when different admixtures are used and also the importance of a correct grout selection for suitable grout injectability. The results summarised in this study are part of a larger study and precede the analysis of the performance of those grouts when injected into different porous media that simulate old masonries walls.

Keywords: *Hydraulic lime grout, Fresh properties, Admixtures*

1. INTRODUCTION

Heritage or common old buildings represent the large majority of the construction types in many urban centres all over Europe. Their masonries frequently present a bad state of conservation and present very different characteristics; some are made of a single leaf, while others have multi-leaf walls. In the case of a multi-leaf walls, the section is composed by two resistant external leaves and an internal space filled by small stones, sand, mortar or other kind of unbounded material [1]. The absence of cohesion among masonry elements, the existence of voids and cracks as well as the poor connection between leaves lead to masonries with non-monolithic behaviour. This means that the wall becomes brittle, namely under vertical and horizontal loads. In order to stabilize such walls and to prevent structural failure grout injection is a current consolidation technique.

The grout injection allows the increase of masonry compactness and creates bonds between the internal and external leafs, therefore improving the masonry mechanical strength and monolithic behaviour. Grouts for injection purpose should be adequately designed to achieve the best performance from the injectability point of view. This means that fresh grout properties, such as

[1] PhD Student, Department of Civil Engineering, Universidade Nova de Lisboa, luis.baltazar@fct.unl.pt
[2] PhD, Department of Civil Engineering, Universidade Nova de Lisboa, fh@fct.unl.pt
[3] PhD Student, Department of Civil Engineering, Universidade Nova de Lisboa, fjorne@fct.unl.pt

rheological properties, seem to be as important as the ones in the hardened state, since the good rheological properties is an essential criterion to allow the correct flow of the grout inside the masonry to ensure the filling of the voids. Thus, the first step for grout optimization for masonries injection should focus on the effects of different grout compositions on its fresh properties. Grouts by definition are mixes of binder and water. However those simple mixes are unable to perform an efficient consolidation, therefore requiring the use of additives, such as superplasticizers, silica fume, fly ashes and air entraining agent, whose efficiency has been consistently shown in literature for the case of cement based mixes [2-4].

This study concerns the use of hydraulic lime grouts with different types of organic and inorganic admixtures. The choice of the hydraulic lime as binder is a consequence of its chemical and physical properties being closer to those of the pre-existing materials in old masonries [5]. So far admixtures like superplasticizer, air entraining agent, silica fume and fly ash have been widely used in the concrete industry. However, little information is presently known regarding the effect of these admixtures in fresh behaviour of hydraulic lime grouts. The main goal of this work is to study the effect of each admixture on the fresh properties of hydraulic lime grouts, such as rheological parameters (as yield stress and plastic viscosity), water retention and stability. Then a selection is made between the ones whose contribution is more relevant to the improvement of the grout performance. The results summarised in this study are part of a larger study and precede the analysis of the performance of those grouts when injected into different porous media that simulate old masonries walls.

1.1. Literature Review about Admixtures

Fly ash is a by-product of coal burning from thermal electric power station, a very fine powder with pozzolanic properties that can react with calcium hydroxide ($Ca(OH)_2$) resulting from hydration of hydraulic lime [2]. According to other researchers [4,6] it is expected that the partial replacement of hydraulic lime by fly ashes contribute to improve the grout fluidity, since the small and spherical fly ash particles will improve the contact between the hydraulic lime particles by ball bearing action and reduce the friction forces.

The silica fume is an inorganic admixture from electrometallurgy industry and it is applied as fine filler whose particles fill into the spaces made by large and long shape of hydraulic lime particles. Due to very fine silica fume particles it is expected a decrease of frictional forces of the system and a reduction of yield stress [7]. Moreover, it is expected that high silica fume fineness increases the absorption of water, thus allowing better water retention capabilities.

The air entraining agents are commonly used in concrete and mortars that will be exposed to liquid water, since they increase the quantity of entrapped air bubbles by reducing surface tension of water (acting as a surfactant material). On the other hand, it is well kwon that rheological properties are directly affected by the air-void system. Thus, the use of air entrained agents may improve the fluidity of cementitious based mixes through small and well distributed air bubbles [8, 9]. In addition, the adsorption of the small air bubbles on the surface of the particles will prevent its skiing and consequently may improve the grout stability through the reduction of segregation.

The superplasticizer is an organic admixture whose action is based on repulsive forces; this means that particles flocculation is reduced or even prevented. The repulsion action contributes to an increase of the distance between particles. So, the addition of a superplasticizer results in a lower segregation and leads to improvement of rheological parameters, through reduction of plastic viscosity and yield stress [10].

2. EXPERIMENTAL DETAILS

2.1. Material Studied

The experimental program was carried out using grouts made with hydraulic lime. The hydraulic lime used is a EN459-1:2010 NHL5 produced in Portugal by Secil-Martingança. The fly ash (FA) used was collected from a Portuguese thermal power plant using coal-fired boilers. A commercially available silica fume (SF) was used, namely undensified silica fume produced by MAPAI. The superplasticizer used was a polycarboxilate (Fluid Premia 180) produced by CHRYSO. A commercially available air entraining agent (AEA) was also used (Sika Aer-5) produced by SIKA.

2.2. Grout composition

The grouts were prepared with a fixed water/binder ratio of 0.5. The inorganic admixtures (FA and SF) were blended with the hydraulic lime at replacement ratio of 10% by lime weight. The grout mixes were prepared with and without the addition of organic admixtures (SP and AEA) at a dosage of 0.5% by weight of hydraulic lime. A total of nine different grout mixes were performed as showed in Table 1.

Table 1 Grout mix compositions

Notation	Constituents	SF (wt%)	FA (wt%)	SP (wt%)	AEA (wt%)	water/binder (-)
Mix 1	Lime	-	-	-	-	
Mix 2	Lime + SF	10	-	-	-	
Mix 3	Lime + FA	-	10	-	-	
Mix 4	Lime + SP	-	-	0.5	-	
Mix 5	Lime + SP + SF	10	-	0.5	-	0.5
Mix 6	Lime + SP + FA	-	10	0.5	-	
Mix 7	Lime + AEA	-	-	-	0.5	
Mix 8	Lime + AEA + SF	10	-	-	0.5	
Mix 9	Lime + AEA + FA	-	10	-	0.5	

2.3. Mixing procedures

The hydraulic lime mixes were prepared at a room temperature of 20 ±2ºC and a relative humidity of 60 ±2%. For the preparation of grouts ordinary tap water was used and both the inorganic admixture (SF or FA) and dry hydraulic lime were hand mixed to ensure a homogeneous distribution before the beginning of the mechanic mixing. Then, the whole powder (binder + inorganic admixture) is added to 70% of total mix water and mixed during 10 min. The delay of 10 min in the addition of SP makes their performance more effective because at early stages of hydration (first minutes) a large amount of hydrates is generated very rapidly (C_3A). If SP is present, they become partly intercalated (co-precipitation) between layers of these hydrates, thus not exerting dispersing action [11]. 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 min at 800 rpm. The blade used for the mixes had a helicoidally shape as shown in Fig. 2. At the end of mixing, each grout sample was passed through a sieve with 1.18 mm (no. 16 ASTM) before the experimental measurements.

2.4. Rheological measurements

Rheological proprieties were evaluated with a Bohlin Gemin HR^{nano} rotational rheometer (Fig. 1), equipped with a plate-plate geometry (with Ø = 40 mm) and a gap of 2 mm. The grout samples were analysed 10 min after the mixing process had ended. In all measures, a pre-shearing stage of 30 s at shear rate of 1 s^{-1} followed by 60 s at rest was applied. Then, the shear rate was increased from 0 to 300 s^{-1} (the maximum shear rate used). For the lowest shear rates (below 25 s^{-1}), their increments were not linear, i.e. each shear rate has been applied long enough until the steady state was reached. However, the total test time (without taking into account the pre-shear) was 2 min. All grout samples were analyzed with a constant temperature of 20ºC, maintained by means of a temperature unit control.

Fig. 1 Rotational rheometer

Fig. 2 The mixer blade used in experimental work

As mentioned above the fresh grout properties can be used as a control factors to know if a grout is suitable to be injected, since a smaller yield value and plastic viscosity means an easier injection process with lower pressures. Based on previous tests it is known that the rheological behaviour of fresh hydraulic lime grouts is a shear-thinning behaviour i.e. decreasing of viscosity with increasing of shear rate [12,13]. Knowing the behaviour of hydraulic lime grouts as a shear-thinning fluid, the modified Bingham model was used to fit the experimental data in order to estimate the plastic viscosity and yield stress [14]:

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

where τ is the shear stress (Pa), τ_0 is the yield stress (Pa), η_p is the plastic viscosity (Pa.s), $\dot{\gamma}$ is the shear rate and c is a constant with no relevant meaning.

2.5. Stability

Grout stability is an essential characteristic to obtain good injectability. Instability phenomena, like segregation and bleeding, cause lower homogeneity in the grout during and after injection. Thus, with an unstable grout the binder particles tend to sink in injected masonry and the flow slows down. The grout stability was analyzed measuring density variations, a procedure developed by Van Rickstal [15]. 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 grout and the immersed volume was calculated for each test (Fig. 3). 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 cup causing a decreasing of density on the top layers.

2.6. Water retention capability

The water retention capability it is an important property to be assured, since it represents the ability of a grout to retain the mixing water during the injection inside dry and high absorptive masonries. The ability to preserve water within grout suspension for the longest possible time will allow to maintain good rheological behaviour and grout stability in order to insure a successful injection. The measurement of water retention was performed in accordance with the ASTM C941-02 [16] as illustrated in Fig. 4.

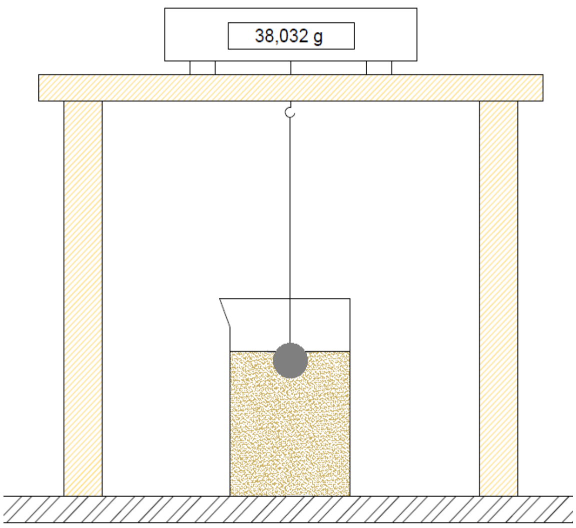

Fig. 3 Configuration adopted for the stability test (adapted from [17])

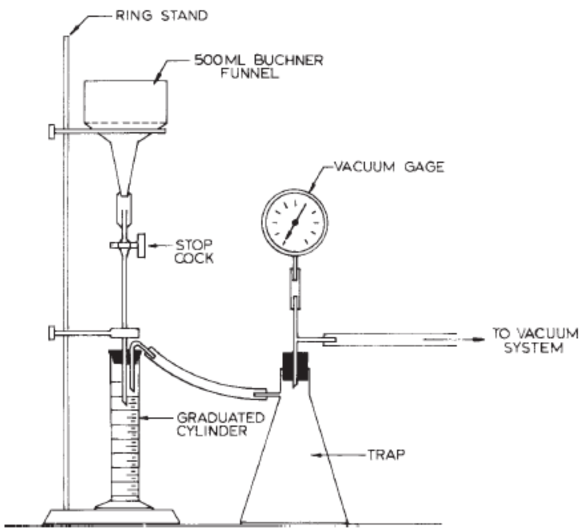

Fig. 4 Device adopted for measurement of the water retention (ASTM C941-02)

3. RESULTS AND DISCUSSIONS

For each grout (from a total of 9 mixes) plots of variation of shear stress as a function of shear rate were analysed. Fig. 5 shows the type of curves obtained from the mean values since at least three repetitions of each mix were performed.

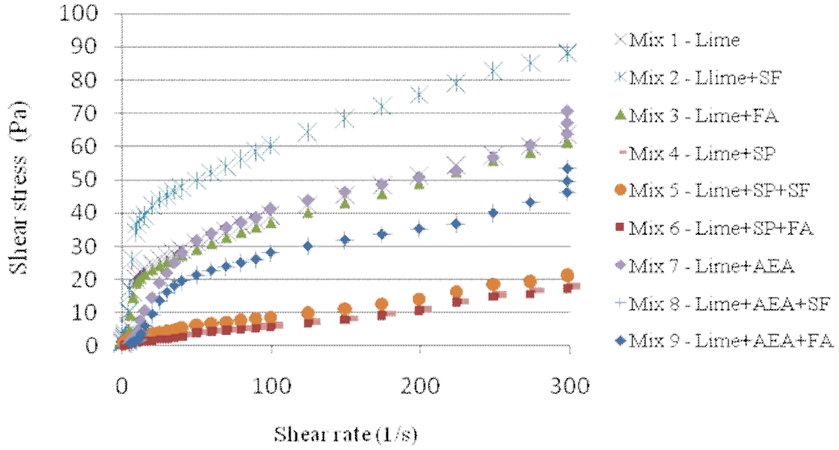

Fig. 5 Shear stress versus shear rate for the nine grout mixes

The yield stress and plastic viscosity values (Table 2) were calculated from the fitting of eq. 1 on the flow curves (as described in the procedure 2.4).

Table 2 Rheological properties results

		Yield stress (Pa)	Plastic viscosity (Pa.s)
Mix 1	Lime	12.74	0.132
Mix 2	Lime + SF	21.49	0.158
Mix 3	Lime + FA	11.63	0.131
Mix 4	Lime + SP	0.470	0.057
Mix 5	Lime + SP + SF	2.060	0.061
Mix 6	Lime + SP + FA	0.310	0.056
Mix 7	Lime + AEA	8.870	0.160
Mix 8	Lime + AEA + SF	19.29	0.190
Mix 9	Lime + AEA + FA	6.340	0.140

The following figures present the effect of each admixture in the grout stability and water retention for each mix was tested (Fig. 6 and 7). Test results are evaluated and discussed below.

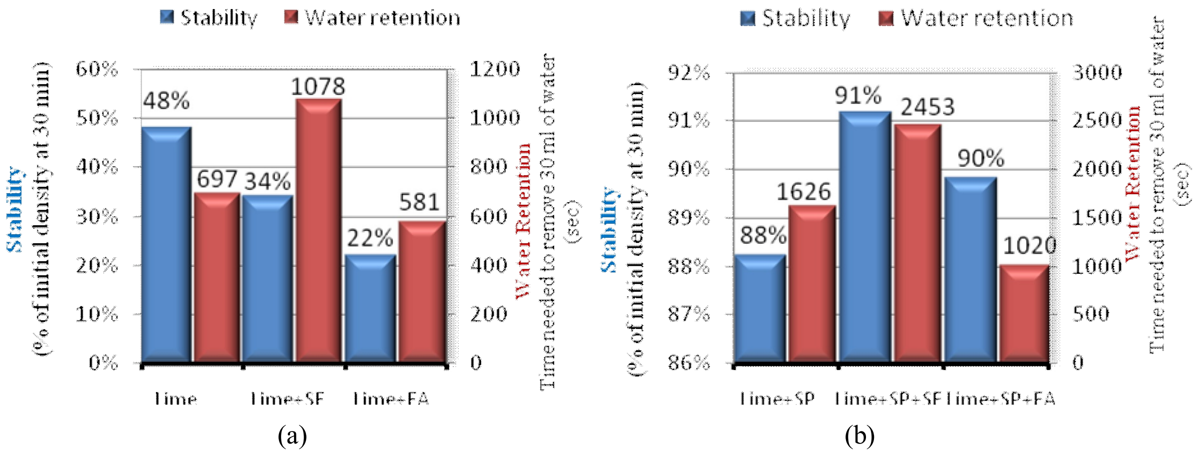

Fig. 6 Effect of admixtures such as SF; FA and SP on stability and water retention of the grouts

From the results analysis (Table 2) it is clear that SF has a harmful effect on the grout rheological behaviour (Mix 2). When the hydraulic lime is replaced by SF, which is an ultrafine powder whose particle dimension is at least 100 times smaller than hydraulic lime particle size, the SF particles will fill the voids between lime particles that results in a suspension with dominant internal friction between closely-packed particles, that cause a reduction on the grout stability as shown in Fig. 6a. On the other hand, the addition of SF leads to an increase of specific surface, causing a higher absorption of water and less free water to bleed, thus a better water retention it is achieved (Fig. 6a). However, the consumption of mixing water by SF results in the increase of yield value and viscosity (Table 2). According to the conclusions of Kadri et al [18] the use of SF causes difficulties in workability of cementitious mixes, requiring the presence of other admixtures such as SPs to solve this problem. Indeed, when SP is added (Mix 5) the fresh grout properties, such as yield value and plastic viscosity, decrease compared to the grout without SP. However, the decreased observed is less significant relatively to the grout with only SP (Mix 4). Even so, the presence of SF together with the SP allows a significant improvement in the grout stability as shown in Fig. 6b.

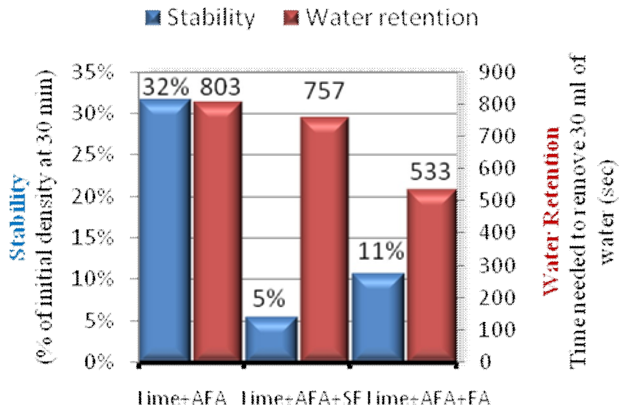

Fig. 7 Effect of admixtures such as SF; FA and AEA on stability and water retention of the grouts

The hydraulic lime replacement by FA results in a slight decrease of the rheological properties. This behaviour is probably due to the replacement of irregular hydraulic lime particles by smaller and spherical morphology FA particles [19], which contribute 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 the mixes 1 and 3. The FA does not adversely affect the rheological performance as it happens with SF, because the FA particles are smaller than SF particles. So, for the same replacement percentage the FA has a lower surface area, thus resulting in a less adsorption of SP or AEA and consequently lower water consumption. That fact leads to an improvement of grout injectability through a slight reduction of yield stress and viscosity values. However, the FA does not produce any improvement in the stability and water retention capability, as shown in Fig. 6a.

From the results of rheological parameters (Table 2) it is clear that SP is the admixture that most contributes to a better grout rheological performing (Mixes 4, 5 and 6), since SP imposes repulsive forces that prevent the solid particles from flocculation, resulting in a larger dispersion of the grout and a decrease of yield stress and plastic viscosity. However, the behaviour of grouts with SF (Mix 5) becomes worse, following the trend of poor performance. Once again this may be due to the effect of a very high specific surface area caused by the SF fineness. On the other hand, 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 from FA particles and dispersing action of SP. As expected the use of the SP also improves the stability and water retention regardless of the presence of SF or FA, as can be seen by comparing the results of the Fig. 6a and 6b. The influence of SP on the stability and water retention is clear, resulting from several phenomena, such as: (i) the dispersing action of SP opposes the sedimentation, so the sedimentation process occurs more slowly and the particles settle more homogeneously; (ii) the SP de-flocculate the grout particles, allowing a higher degree of wettability and consequently reducing the amount of free water [20].

The rheological properties are affected by the air voids inside the grout microstructure. Hence, the small air bubbles, which work as a deformable inert in the mixture, help to disperse the hydraulic lime particles [8]. The AEA provides the production and fixation of small air bubbles (smaller than lime particles) on the particle surface that improve the grout fluidity, owing to its ball bearing effect and without the drawback of water consumption as it happens with SF or FA. That fact leads to a considerable decrease of yield stress for grout with AEA (Mixes 7, 8 and 9) when compared to the grouts without any organic additive (Mixes 1, 2 and 3). However, from a stability and water retention point of view (Fig. 7) the use of AEA leads to the worse gout performance, since it got lower stability than in all the other mixes. Moreover, its performance is further worsened with the presence of SF or FA; this is due to an increase in surface area that enhances the amount of admixture adsorbed on particles surfaces. Therefore, a less amount of AEA molecules are available for the air bubble formation [8]. On the other hand, it can be seen in Fig. 7 that a high water retention capability at very low stability values was obtained (mixes with SF and FA). This may be due to 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 2.6).

4. CONCLUSIONS

In the light of the achieved results, it is clear that SF has a harmful effect on the rheological behaviour of the hydraulic lime grouts and requires the presence of SPs to solve this problem. On the other hand, FA does not adversely affect the rheological performance, since it 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). However, the FA does not produce any improvement in the stability or water retention capability. In contrast the SP is the admixture that most contributes to a better grout performance, since it imposes repulsive forces that prevent the solid particles from flocculation, resulting in higher fluidity, stability and water retention capability. From the rheological measurements the AEA leads to a decrease of yield stress. However, from stability and water retention point of view (Fig. 7) the use of AEA leads to the worse gout performance.

Thus from a comprehensive point of view considering rheological, stability and water retention results it seems that products like SP are inevitable when designing a grout for injection purpose and its presence seems to be particularly required when other powder admixtures are used (such as SF). The authors recognize that these results should not be generalized, since the influence of the analyzed admixtures will depend on other parameters that have not been considered in this study, such as different hydraulic lime and water/binder ratio. However, this investigation demonstrated the potential of some admixtures in the improvement of fresh properties of hydraulic lime grouts and precedes the grout composition optimization and also the analysis of the performance of those grouts when injected into different porous media that simulate old masonries walls.

ACKNOWLEDGEMENTS

This paper is part of the research project PTDC/ECM/104376/2008, funded by FCT/MCTES, Portugal. The authors would like to acknowledge the hydraulic lime and the superplasticizer provided by *Secil-Martingança* and *Chryso – Chemicals Solution for Construction Industry*, respectively and Mr. Victor Silva who contributed to materials preparation.

REFERENCES

[1] Vintzileou E. (2011). Three-leaf masonry in compression, before and after grouting: a review of literature. *International Journal of Architectural Heritage* 5: 513-38.

[2] Rudzirnski L. (1984). The effect of fly ashes on the rheological behaviour of cement pastes. *Materials and Structures* 17(5) 369-373.

[3] Demir I, Baspinar M.S. (2008). Effect of silica fume and expanded perlite addition on the technical properties of the fly ashlime–gypsum mixture. *Construction and Building Materials* 22: 12991304.

[4] Chindaprasirt P, et al. (2005). Effect of fly ash fineness on compressive strength and pore size of blended cement paste. Cement & Concrete Composites 27: 425-28.

[5] Binda L, et al. Repair and investigation techniques for stone masonry walls. *Construction and Building Materials* 11(3): 133-42.

[6] Chindaprasirt P, et al. (2005). Mixed cement containing fly ash for masonry and plastering work. *Cement & Concrete Composites* 17: 612-18.

[7] Lasker A.I, Sudip T. (2008). Rheological behavior of high performance concrete with mineral admixtures and their blending. *Construction and Building Materials* 22: 2345-54.

[8] Du L, Folliard K.J. (2005). Mechanisms of air entrainment in concrete. *Cement and Concrete Research* 35: 1463-71.

[9] Ouyang X, e tal. (2008). The feasibility of synthetic surfactant as an air entraining agent for the cement matrix. *Construction and Building Materials* 22: 1774-79.

[10] Grzeszczyk S, Front K. (2000). Effect of superplasticizers on the rheological properties of fly ash suspensions containing activators of the pozzolanic reaction. *Cement and Concrete Research* 30: 1263-66.
[11] Flatt R.J, Houst Y.F. (2001). A simplified view on chemical effects perturbing the action of superplasticizers. *Cement and Concrete Research* 31: 1169-1176.
[12] Bras A, et al. (2010). Effect of environmental temperature and fly ash addition in hydraulic lime grout behaviour. *Construction and Building Materials* 24:1511-7.
[13] Roussel N, et al. (2010). Steady state flow of cement suspensions: A micromechanical state of the art. *Cement and Concrete Research* 40:77-84.
[14] Barnes H.A. (2000). A handbook of elementary rheology. *Institute of Non-Newtonian Fluid Mechanics*. University of Wales.
[15] Rickstal, F.V. (2000). Grout injection of masonry, scientific approach and modeling. PhD thesis. KU-Leuven.
[16] ASTM Designation C938-02 (2002) Standard pratice for proportioning grout mixtures for preplaced-aggregate concrete.
[17] Bras A, Henriques F.M.A. (2009). The influence of the mixing procedures on the optimization of fresh grout properties. *Materials and Structures*: 42:1423-32.
[18] Kadri E-H, Aggoun S. (2009). Interaction between C_3A, silica fume and naphthalene sulphonate superplasticiser in high performance concrete. *Construction and Building Materials* 23: 3124-8.
[19] Lasker A.I, Sudip T. (2008). Rheological behavior of high performance concrete with mineral admixtures and their blending. *Construction and Building Materials* 22: 2345-54.
[20] Mikanovic N, Jolicoeur C. (2008). Influence of superplasticizers on the rheology and stability of limestone and cement pastes. *Cement and Concrete Research* 38: 907-19.