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EFFECT OF HYDROPHOBIC SILICA FUME ON THE RHEOLOGICAL BEHAVIOUR OF INJECTION GROUTS

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ABSTRACT

The consolidation of old masonry buildings has emerged as a need, due not only to structural strengthening but also to conservation of the architectural heritage. The grout injection is a technique used for the consolidation and strengthening of old masonry walls. The grout, once injected into the internal structure of the masonry walls, enhances the cohesion of the wall by filling the empty spaces and cracks in the masonry. The use of pozzolans like silica fume in injection grouts has become of vital importance from technical and sustainability point of view. Silica fume plays an important role in the production of more durable grouts with improved mechanical characteristics. Nevertheless, various authors point out some drawbacks regarding the use of silica fume in cementitious-based mixtures. Among these, the loss of workability during the fresh state is the most harmful. The present research aims to study the effect of a new-silica fume with hydrophobic properties on the rheological properties of injection grouts in comparison to that of an ordinary silica fume. The experiments were conducted using different silica fume dosages with hydraulic lime-based grouts proportioned with a polycarboxylate-based high range water reducer. The experimental results revealed that grouts with hydrophobic silica fume had better rheological behaviour (lower consistency and yield stress) and, therefore, an improved flowability, even at high dosage of silica fume.

1. INTRODUCTION

The overall goal of this research is to study the effect of a new-silica fume with hydrophobic properties on the rheological properties of injection grouts for consolidation of old stone masonry walls. Stone masonry buildings are one of the most common construction types in many European cities centres. Those masonry walls often need consolidation to improve their mechanical performance. Old stone masonry walls are usually characterized by their constructive weaknesses, such as scarce or no connection through the thickness and presence of voids and cracks which lead to instability of the masonry under both horizontal as well as vertical loads. The consolidation of stone masonry buildings are amongst the most important building conservation activities. This is so, because besides safeguarding the human lives involved, the cultural heritage has to be preserved. The grout injection is a technique currently used for the consolidation and strengthening of old stone masonry walls. Grout injection is a process of introducing a fluid mixture into the masonry for the consolidation and improving of mechanical properties of the whole masonry system. Grouts can be represented as biphasic system consisting of suspended particles (binder) in a continuous fluid phase (water). The efficacy of the grouts depends mainly on the injectability properties, rather than its hardened characteristics. Therefore it is essential to evaluate the effectiveness of the injection grouts in terms of keeping their properties that are considered as adequate. For that purpose the use of rheology has proven to be useful. So, this research will focus in depth the relations between grout rheological properties and silica fume type and dosage.

Silica fume is one of the most used pozzolanic materials. Because of its extreme fineness and high silica content, silica fume is a highly effective pozzolanic material. Silica fume is a by-product of the electrometallurgy industry, which has been mainly used as a binder replacement in order to reduce the binder content usually for economic reasons and global climate protection. It was first used in 1969 in Norway but only began to be systematically employed in North America and Europe in the early 1980s. Since then, the use of silica fume (manly in concrete) has been increasing rapidly. The rapid increase in the use of silica fume is attributed to its positive effects on the properties of the cementitious materials, such as increase of compressive strength, increase of the pack density and thus elimination or minimization of bleeding. Several studies (Shannag 2000, 2002; Wei-Hsing 1997) have shown that the properties of cementitious materials can be improved when silica fume is used and, specially, when it is combined with other admixtures such as high range water reducers (HRWR). HRWRs are water-soluble organic chemicals, which are used to improve the fluidity of mixtures by dispersing binder and additives particles present in the pastes.

Previous studies involving silica fume and hydraulic lime have shown that silica fume has a harmful effect on grout rheological behaviour, causing an increase of yield stress and plastic viscosity, which results in a grout with inferior injectability (Park et al. 2005; Baltazar et al 2013, 2014a, 2014b). Based on those results and knowing that the decreasing of grout workability is associated with the higher specific surface of silica fume particles, the authors developed a silica fume with hydrophobic properties in order to improve the grout flowability, especially at high silica fume dosage. The aim of the present research is to evaluate the influence of replacement of hydraulic lime by a silica fume with hydrophobic properties on the rheological properties of grouts for old masonry consolidation.

2. MATERIALS AND METHODS

The choice of hydraulic lime as binder is a consequence of its compatibility with pre-existing materials in old masonries. According to other authors (Binda et al. 2006), who studied the compatibility of materials used for repair of old masonry buildings, the compatibility between the grout and the original materials should be ensured in three aspects: chemical compatibility, mechanical compatibility and historical compatibility. Lime based binder are widely agreed to be the most compatible material for restoring of heritage buildings (Ferragni et al. 1982, Ballantyne 1996). This is because lime present higher ductility and its properties regarding to moisture transport and thermal expansion, among others, are closer to those of the original materials than, for instance, the Portland cement. The hydraulic lime used is EN459-1:2010 NHL5 produced by Secil-Martingança (Portugal). Polycarboxylate-based HRWR (labelled as Glenium sky 617) produced by BASF was used in the conducted research. The silica fume used was an undensified silica fume produced by Mapei (Portugal).

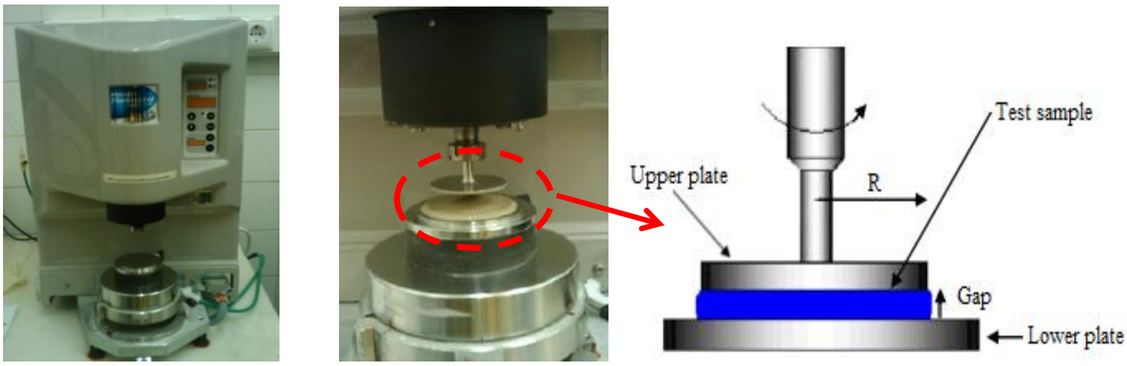
A comparison between two types of silica fume was made, namely ordinary silica fume (as provided by the producer) and the one with hydrophobic treatment. In order to carry out the hydrophobic treatment the following procedure was carried out: first, silica fume was deep impregnated with a hydrophobic solution (i.e. solution of a water-soluble siliconate). Then, the impregnated silica fume was placed in the climate chamber in order to evaporate the solvent of the hydrophobic substance. Finally, the dry silica fume was sieved and blended in order to get a homogeneous powder.

Different dosages of silica fume were tested for both types of silica fume (as replacement of hydraulic lime), keeping constant the HRWR dosage and water/binder ratio, as shown in table 1. The HRWR dosage and water/binder ratio were proportioned in order to highlight the data that can result from such grouts. The grout no. I (control grout) was designed without any type of silica fume.

Table 1 Compositions of tested NHL grouts

No.	Amount of lime (%)	Silica fume (wt%)		HRWR (wt%)	w/b ratio
		Ordinary	Hydrophobic		
I	100	-	-	0.8	0.5
II	90	10	-	0.8	0.5
III	70	20	-	0.8	0.5
IV	40	30	-	0.8	0.5
V	90	-	10	0.8	0.5
VI	70	-	20	0.8	0.5
VII	40	-	30	0.8	0.5

Rheological properties were evaluated with a Bohlin Gemini HR^{nano} rotational rheometer, equipped with a plate-plate geometry (with Ø = 40mm) and a gap of 2mm (see Fig. 1). 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 60s at shear rate of $1s^{-1}$ followed by 60s at rest was applied. The pre-shearing of 60 deformation units was applied, before starting the rheological measurements, in order to homogenize the sample and to eliminate its shear history because of thixotropic character of cementitious materials. Then, the shear rate was increased from 0 to $100s^{-1}$. Each shear rate was applied long enough in order to ensure the attendance of the steady state, before measurements took place. All grout samples were analysed with a constant temperature of 20ºC, maintained by means of a temperature unit control.



(a) (b)

Fig. 1 Illustration of (a) rotational rheometer and (b) plate-plate geometry with sample loaded

The hydraulic lime grouts were prepared at room temperature of 20±2ºC and a relative humidity of 60±2%. For the preparation of grouts ordinary tap water was used. The silica fume was added and mixed with the dry hydraulic lime separately, to ensure a homogeneous distribution, before being transferred to the mechanical mixer. The mixture procedure adopted was the one achieved on a previous study (Baltazar 2012): the whole powder (lime + silica fume) is added to 70% of total mix water and mixed for 10 minutes. The remaining water (with diluted HRWR) is added within 30s (without stopping the mixer). After all materials had been added, the mixture was maintained for an additional 3 minutes at 800 rpm.

3. RESULTS

Rheological properties of injection grouts are decisive parameters since they affect the fresh performance and therefore the durability of hardened grout. Thus, the influence of silica fume type and dosage on rheological properties of admixtures NHL-based grout was first characterized. The flow curves of hydraulic lime grouts as function of silica fume dosage are presented in Fig. 2. The rheograms (Fig. 2) show that the flow curve of natural hydraulic lime grouts with and without silica fume is not totally linear, but has an initial linear behaviour that is followed by a non-linear flow (shear-thinning behaviour), where the rheological behaviour can be modelled fairly well using the Herschel-Bulkley model or the modified Bingham model (Barnes 2000). In this study, the Herschel-Bulkley model was used to describe grout rheological behaviour:

$$\tau = \tau_0 + K \times \dot{\gamma}^n \qquad (1)$$

where: τ is the shear stress (Pa), τ_0 is the yield stress (Pa), *k* is the consistency coefficient (Pa.s^n), $\dot{}$ is the shear rate (s^{-1}) and *n* is the fluidity index which characterizes shear-thinning behaviour of grout. Experimental data were fitted with the Herschel-Bulkley model independent of silica fume type and dosage. Then, the three Herschel-Bulkley parameters were chosen to compare grouts to each other.

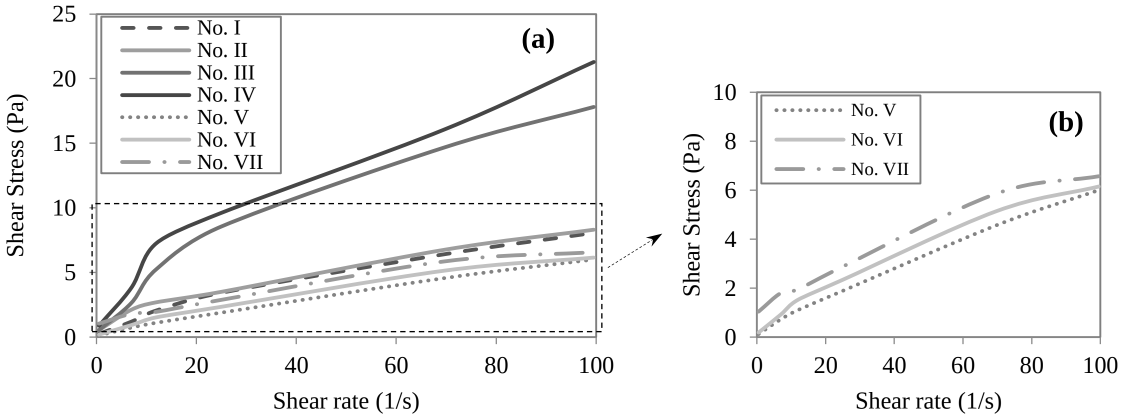


Fig. 2 (a) Shear stress versus shear rate in NHL grouts of different composition; (b) focus on grouts with hydrophobic silica fume

From a practical point of view, yield stress is associated with the minimum stress that is necessary to apply for the suspension to start flowing. The knowledge of the yield stress enables to understand if a fluid will flow or not, since it represents that threshold. This important property affects the flow behaviour of the grout and its capacity to flow inside a porous medium (masonry inner core). On the other hand, consistency coefficient is associated to grout's viscosity, since an increase of the consistency coefficient leads to a grout viscosity increase. So, a low consistency means that the grout flows easily whereas for high consistency coefficient the flow will be much more difficult.

3.1 Yield stress and consistency

Figs. 3 and 4 present the evolution of the yield stress and consistency coefficient according to the silica fume used and its dosage (as partial replacement of lime in weight percentage). A smaller yield stress and consistency is better than a greater one. From the rheological parameter results it is clear that as the ordinary silica fume dosage increases, the grout becomes less workable. Even for the lowest dosage of silica fume (10wt%) it can be observed that yield stress and consistency increased regarding the reference grout (No. I). The addition of silica fume leads to an increase of specific surface, causing higher adsorption of mixing water resulting in a decrease of grout flowability and therefore a lower injectability (Toumbakari 2002). This behaviour is consistent with the observations of Park et al. (2005) and Baltazar et al (2013) that flow resistance steeply increases with increasing silica fume dosage (as binder replacement). This behaviour is dependent on the solid volume fraction, meaning that when the silica fume dosage exceeds a threshold value, and according with the Krieger-Dougherty equation, an increase in the grout viscosity occurs (Struble and Sun 1995; Phan et al 2006). In other words, the introduction of small-sized silica fume particles is the source of additional surface area resulting in an increase of contact forces among fine particles and therefore they coagulate very easily due to interparticle interactions (Van-der-Waal's interactions). The penetration capacity of such grouts is significantly decreased, making its injection at low pressure rather difficult. Thus, for a required penetrability performance, the water content of the grout must increase, with detrimental effects on the fresh grout stability and on the mechanical properties of the hardened grout.

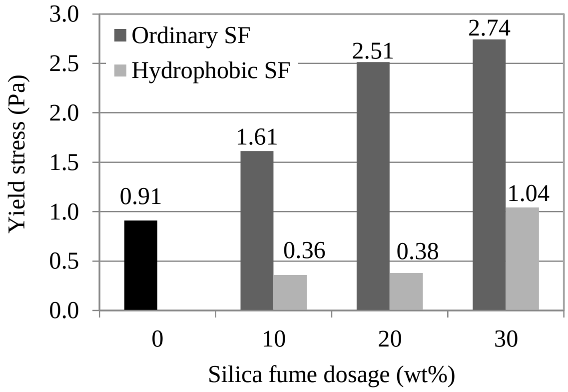


Fig. 3 Evolution of the yield stress (τ_0) according to the dosage and the silica fume studied

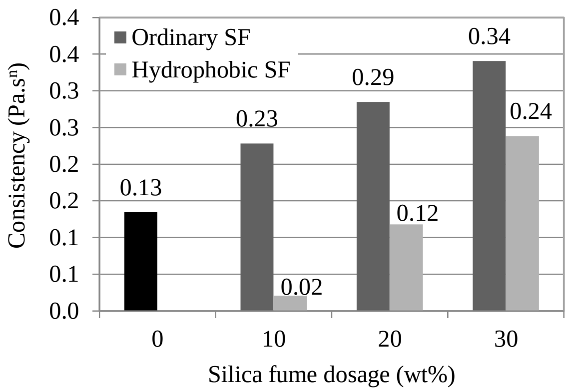


Fig. 4 Evolution of the consistency coefficient (*k*) according to the dosage and the silica fume studied

As mentioned, a good flowability (i.e. grouts with low consistency and yield stress) is preferred for injection purposes. The results illustrate that the rheological properties of grouts containing silica fume with hydrophobic treatment are significantly improved compared to the grouts containing ordinary silica fume. It is worth mentioning that grouts containing 20wt% of hydrophobic silica fume were found to have lower rheological parameters than the grout without any silica fume (composition I). Thus, it can be stated that the hydrophobic properties of silica fume can be useful to improve the rheological performance of injection grouts. This is due to the fact that that hydrophobic silica fume acts as fine filler (with reduced absorption of water) whose small-spherical shape particles (Fig. 5) fill the spaces made by the large and the long shape particles of hydraulic lime binder imposing a ball bearing effect, which reduce the friction forces between lime particles and therefore an improvement of grout flowability is obtained.

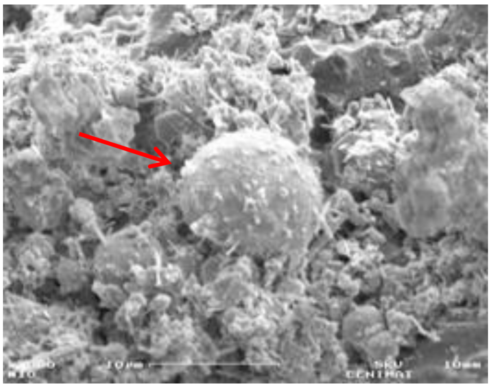


Fig. 5 SEM image illustrating the spherical shape of hydrophobic silica fume particle

3.2 Fluidity index

Fig. 6 shows the evolution of the fluidity index *n* calculated from the Herschel-Bulkley model according to the silica fume used and its dosage. The rheograms (Fig. 2) show that most of the grout compositions tested presented a shear-thinning behaviour, which means that *n* is lower than 1. A shear-thinning behaviour means that if flow velocity decreases during grout injection it leads to a viscosity increase (which is not desirable), thus *n* value should be closer to 1 in terms of injection grouts.

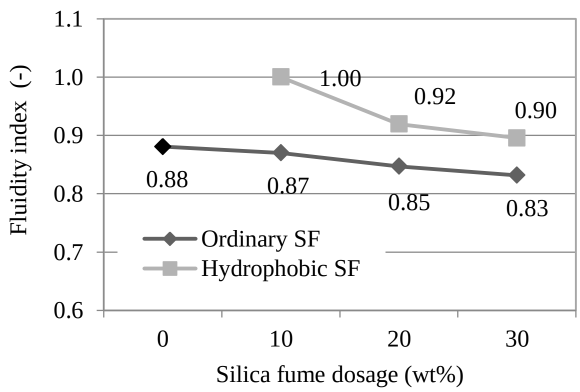


Fig. 6 Evolution of the fluidity index (*n*) according to the dosage and the silica fume studied

For the grouts tested when the dosage of silica fume increases, the fluidity index decreases. Indeed, the higher adsorption of water by silica fume explains this decrease.

However, grouts with hydrophobic silica fume, fluidity index are always higher than the grouts with ordinary silica fume. In fact, for grout containing 10wt% of hydrophobic silica fume, fluidity index was found to be equal to 1 which means a Bingham's behaviour. Baronio et al (1992) carried out an experimental program with masonries of different dimensions with cracks and voids irregularly distributed, which showed that it was difficult to conduct the injections with a constant pressure. A grout exhibiting Bingham's behaviour (achieved with hydrophobic silica fume) enables grout to flow easily inside porous medium, even when velocity decreases during injection, since viscosity will not change.

4. CONCLUSIONS

This paper focuses on the influence of hydrophobic silica fume and its dosage on rheological behaviour of NHL-based grouts. Herschel-Bulkley model (Eq. 1) was used to fit the experimental data. From the results obtained, the following conclusions can be derived:

1. Ordinary silica fume has a harmful effect on grout rheological behaviour, resulting in an increase of yield stress and consistency values.
2. The rheological properties of grouts containing silica fume with hydrophobic treatment are significantly improved.
3. The grout containing 20wt% of hydrophobic silica fume (as replacement of hydraulic lime) showed better rheological performance than the reference grout (without any silica fume).
4. It is believed that the hydrophobic silica fume acts as fine filler, filling the spaces made between the long shape particles of hydraulic lime imposing a ball bearing effect, which improves the grout flowability by reducing the friction forces between lime particles.
5. Silica fume has a strong impact on the shear-thinning behaviour of NHL grouts: the presence of hydrophobic silica fume leads to a more Bingham's behaviour.

This study has showed that the silica fume with hydrophobic treatment can be used to control and improve the rheological properties of hydraulic lime grouts for consolidation of old stone masonries. It is clear, however, that additional characterization of the hardened state properties of grouts containing this type of silica fume are needed, because the hydrophobic behaviour may compromise some benefits of using silica fume, such as its pozzolanic reaction with the free lime to form additional calcium silicate hydrate.

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