Rheology of grouts for masonry injection

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Abstract. Grout injection technique applied in a multiple leaf masonry walls aims at increasing the compactness of the masonry and, therefore, improving their monolithic behaviour by bonding the separated segments together without compromising the architectural value of the masonry building. The success of the injection grouts depends mainly on their rheological properties. This means that the flow properties of fresh grouts are as important as their properties in the hardened state, since they govern the ability of the grout to flow and fill the voids within masonry. In practice, the variability of voids within masonry requires ability to fine tune the rheological properties of the grout, in order to optimal fill all voids with grout. So, models were developed with the main purpose of predict and control the rheological properties of grouts just performing simple flow tests traditionally used in the field. It is well known that flow tests commonly performed (such as Marsh cone and slump test), which try to describe the workability of cementitious materials, only give a qualitative result that can not be expressed in physical rheological units. However, the proposed models allow calculating a physical parameter (e.g. viscosity) instead of an empirical one (e.g. fluidity). This research shows the effect of grout design variables on the properties of natural hydraulic lime grouts based on the correlation between rheometry measurements and simple flow tests. Furthermore, the proposed models can be very useful to smooth the grout design methodology, since these models enable the prediction of rheological properties of the grout without the need for expensive and complex equipment, such as rheometers or viscometers.

Introduction

This paper is based on the study of natural hydraulic lime grouts used for consolidation of old stone masonry walls. The performance of these masonries relies on their monolithic behaviour rather than on the mechanical properties of their individual elements. This means that the good condition of the connections between elements is essential towards a good performance. In general, stone masonry walls, especially those made of multi-leafs, are prone to detach from the rest of the building and to fail out-of-the plane by disaggregating into different parts, for instance under a seismic event [1,2]. A typical stone masonry wall is mainly subjected to compressive stresses. However, a combination of compressive and tensile stresses (i.e. flexural stresses) can also occur and cause very harmful consequences to this type of walls. Flexural stresses are usually induced by seismic vibrations, shocks and wind loads, among others, which may end up by causing the masonry failure. This vulnerability under certain types of loads is due to several factors, such as: absence of cohesion between the external elements i.e. poor transversal bonds between the leaves of the wall and weakness of the inner core. Hence, every consolidation technique should introduce materials capable of withstanding these weaknesses.

Grout injection is one consolidation technique and it is defined as the introduction of a fluid material injected under pressure into the masonry inner core. Injection grouts aim to fill cracks and voids and to create bonds between the delaminated layers of the wall, ensuring the continuity of the repaired masonry. Grouts are mixtures of a binder with water and admixtures, which must feature adequate fluidity and penetrability in order to be injectable. In most cases, the selection of grout materials depends on the desired fresh properties and performance characteristics. It has been pointed out that fluidity, penetration and stability (segregation and bleeding) are essential fresh properties [3,4], meaning that during injection grouts should preserve their fluidity with minimal segregation and clogging. The rheological behaviour of fresh grouts has great importance on their injectability and, consequently, on the quality of the grouting operation. Rheology is often used as a tool in the design of cementitious suspensions, such as cement based pastes, mortars, concretes and grouts [5,6,7]. Rheological measurements allow the evaluation of the effects of different grout composition on the fresh behaviour and a proper characterization through rheological models [8]. Based on previous works [6,9] it is accepted that fresh behaviour of natural hydraulic lime grouts can be represented using the Bingham model. Therefore, if the fresh grout is assimilated to a Bingham fluid two independent parameters are needed to describe the rheological behaviour, such as yield stress and plastic viscosity. From a practical point of view, yield stress is associated with the minimum stress that is necessary for a grout to start flowing. On the other hand, plastic viscosity represents the flow resistance once flow is initiated. A low plastic viscosity means that the grout flows easily whereas for high plastic viscosity the flow will be much more difficult.

Thus, traditional rheology tests using rheometers or viscometers can be used to predict the rheological behaviour of grouts; however these tests are often time-consuming and give too many information when only the yield stress and plastic viscosity are needed [10]. The main objective of this paper is to model the effect of water/binder ratio and dosage of superplasticizer on the rheological properties of natural hydraulic lime grouts using simple tests easy to use at construction sites, such as Marsh cone and spread test using a mini-slump test. The established models allow estimating the rheological properties, meaning that these models are able to calculate a physical parameter (e.g. viscosity) instead of an empirical one (e.g. fluidity). The proposed models can be very useful to smooth the grout design methodology, since the rheological properties of the grout can be predicted just by performing simple flow tests traditionally used on field.

Methodology

The influence and significance of each grout design variable on its rheological behaviour was identified in a previous work [6] using the design of experiments method (DOE) together with analysis of variance (ANOVA). Based on these previous results, the water/binder ratio and dosage of superplasticizer were pointed out as the most significant grout components. These grout components together with the outputs from simple flow tests (Marsh cone and mini-cone test) were used to formulate the mathematical models for prediction of the rheological properties that best characterize the fresh grout performance. The proposed models have a valid domain for grouts made with 100% of NHL, water/binder content of 0.45 – 0.55 and superplasticizer dosage of 0.6 – 1.2%, by mass of lime.

Experimental details

Materials. The experimental program was carried out using grouts made with natural hydraulic lime (NHL) labelled as NHL 5 produced according to the European Standard EN459-1:2010 [11]. A commercially available polycarboxylate-type superplasticizer (Glenium Sky 617) was used.

Mixing procedure. The hydraulic lime grouts were prepared at room temperature of 20±2ºC and a relative humidity of 60±5%. For the preparation of grouts ordinary tap water was used. The mixing procedure adopted was obtained in previous research [4]: the whole lime is added to 70% of total mix water and mixed for 10 minutes. The remaining water (with diluted superplasticizer) 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.

Experimental procedures. Three experimental procedures were performed simultaneously: (i) rheological measurements using a rheometer to evaluate the grouts rheological properties, (ii) Marsh cone test to evaluate the flow time and (iii) mini-slump test to estimate the spread diameter.

For each grout mix, three samples were collected to be used on the rheological measurements, Marsh cone test and mini-slump test. All these experimental tests were performed at 5 minutes after the end of mixing process. Then, the correlation between the rheological properties and the results obtained from both Marsh cone and mini-slump tests were established.

Rheological measurements. The rheological measurements were performed using a Bohlin Gemin HR^{nano} rotational rheometer, equipped with a plate-plate geometry (with Ø = 40 mm) and a gap of 2 mm (see Fig. 1). 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 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 not be 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 $300s^{-1}$. Each shear rate was applied long enough in order to ensure the attendance of the steady state, before measurements were recorded. All grout samples were analysed with a constant temperature of 20ºC, maintained by means of a temperature unit control. A solvent trap was used to prevent drying of the grout samples during testing. The results were interpreted in the frame of rheology suspension knowledge; this means that the Bingham model (Eq. 1) was adopted to describe the grout rheological behaviour and to determine the plastic viscosity [12]:

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

where τ is the shear stress (Pa), τ_o is the yield stress (Pa), η_p is the plastic viscosity (Pa.s), and $\dot{\gamma}$ 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 Eq. 1 in this work the yield stress values were obtained from the correspondent shear stress value obtained at $\dot{\gamma} = 0.5s^{-1}$. According with other research [13] this method of estimating yield stress minimises the error associated with the lack of sensitivity of rheological models at low shear rates.

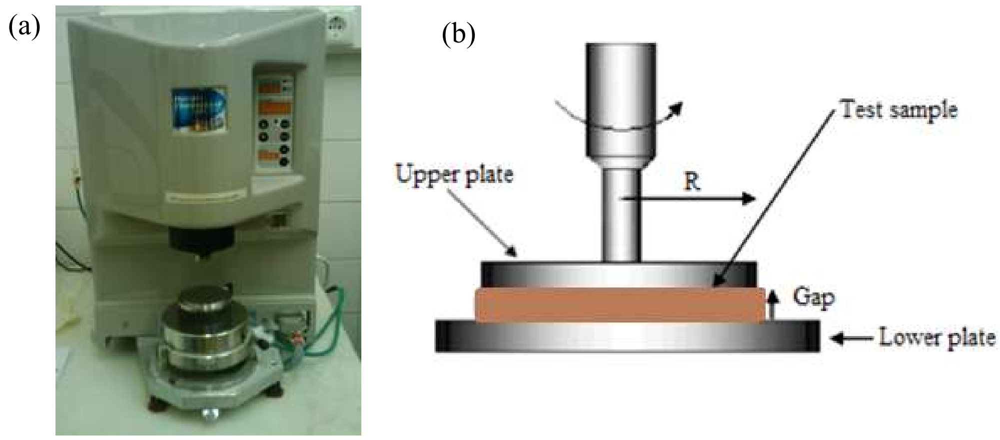

Figure 1. Illustration of (a) rotational rheometer, (b) plate-plate geometry

Marsh cone test. The Marsh cone test was performed according to ASTM C939-02 [14]. Based on this standard the measurement of flow time is connected to the grout fluidity, meaning that the longer the flow time, the lower is the grout fluidity. In this context, the time needed for a grout to flow is proportional to the viscosity of the grout; this means that Marsh cone test can be seen as an index of grout viscosity [10]. The Marsh cone used in this work has a capacity of 1980ml and an opening with 5 mm of diameter (see Fig. 2). A volume of 1,000 ml of grout was placed into the cone and the flow time (expressed in seconds) required for 800 ml of grout to flow out was measured.

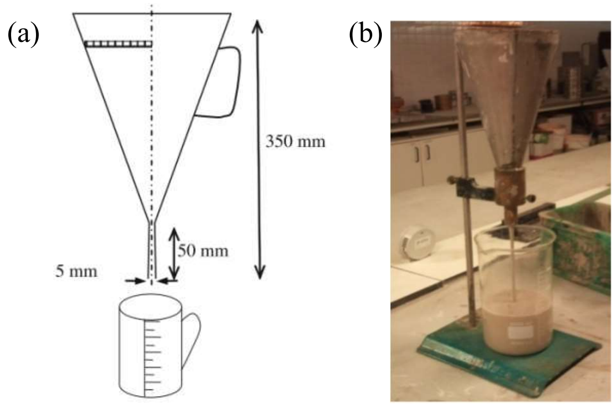

Figure 2. Illustration of (a) Marsh cone dimensions and (b) Marsh cone apparatus

Mini-slump test. The mini-slump test is other common approach to quantify the fluidity of cementitious mixtures. This measurement technique is used extensively for evaluation of self-compacting concrete due to the low yield stress of such materials. In this study the mini-slump test adopted is similar to the procedure developed by Roussel et al [10]. The modus operandi was the following: the cone was laid on a horizontal glass surface. After the careful placing the grout into the mini-cone, to avoid bubbles formation and grout overflow, the cone is vertically lifted. After lifting the mini-cone the grout flows by gravity and the slump occurs (see Fig. 3b). The grout will flow while the local stress is higher than the material yield stress. In all cases, the slumped area was circular, indicating a uniform deformation throughout the grout sample (see Fig. 3c). The spread diameter was measured when the flow stopped. Each spread diameter value is the mean of two measurements along two perpendicular directions. The geometries of the mini-cone used are given in Fig. 3a.

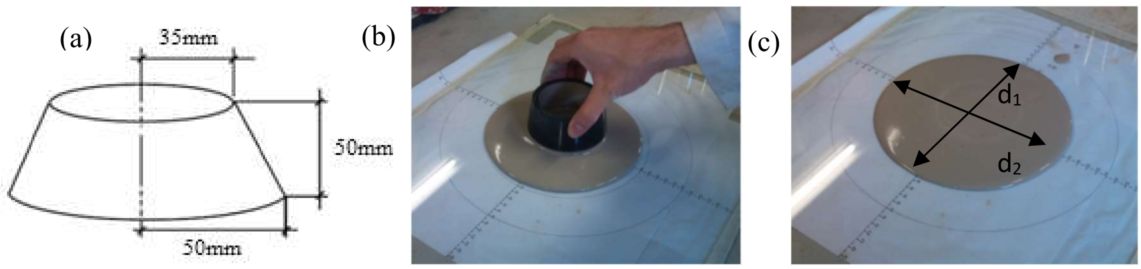

Figure 3. Illustration of (a) mini-cone geometry; (b) lift the mini-cone and (c) spread sample at stoppage

Results and discussion

Rheological measurements. The plastic viscosity values (η_p) were calculated from the fitting of Eq. 1 to the experimental data. NHL grouts have shear-thinning behaviour and only approximate linearity as the particles become deflocculated at high shear rates. Directly measuring the stress required to initiate flow is the best method for determining yield stress. Hence, the yield stress values correspond to the shear stress measured at shear rate of 0.5 s^{-1}.

The effect of superplasticizer dosage and water/binder ratio on grout rheological properties is presented in Fig. 4 and 5. As expected the plastic viscosity and yield stress values decrease with the increasing of superplasticizer dosage. Since it is known that superplasticizers impose repulsive forces that prevent the particle flocculation, higher dosages mean higher capacity of particles dispersion resulting in a larger dispersion of the grout and a decrease of rheological properties [15]. However, it should be noted that for high dosages of superplasticizer (>0.8wt%) only slight improvements on workability were observed. Moreover, it can be seen that the yield stress is more affected with increasing superplasticizer dosage than the plastic viscosity. The water/binder content has a similar behaviour to the superplasticizer dosage; notwithstanding, simple addition of water to make the grout more fluid is an inadequate option because a higher water/binder ratio will weaken the grout in the hardened state and will increase the shrinkage and the free water amount that also contributes to instability phenomena during the fresh state of the grout [16].

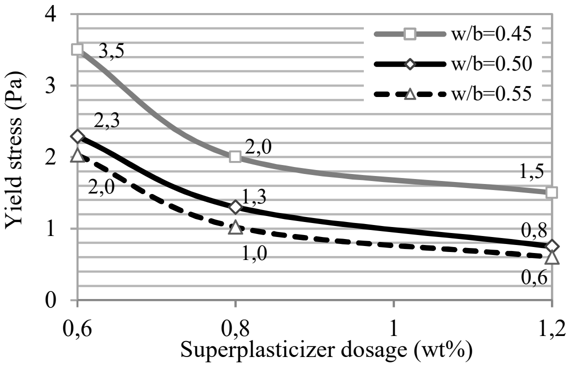

Figure 4. Influence of water/binder ratio and superplasticizer dosage on plastic viscosity

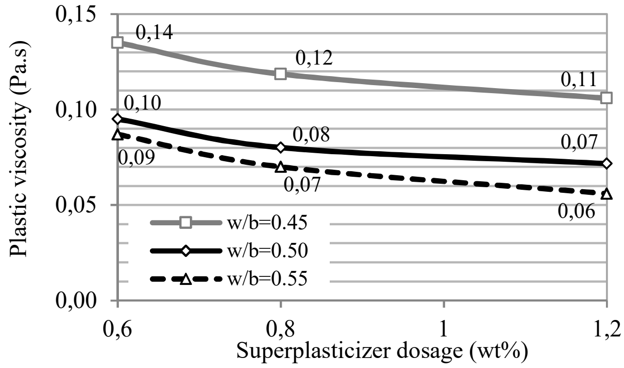

Figure 5. Influence of water/binder ratio and superplasticizer dosage on yield stress

Marsh test and mini-cone test. The Marsh cone test was used to analyze the loss of fluidity of different grout compositions. This test gives the fluidity in terms of the time needed for a grout sample to flow thought the cone, which is proportional to the fluidity of the grout (inversely proportional to viscosity). The Marsh cone test is a simple test which can also be used to achieve the saturation point or optimum dosage of superplasticizer that corresponds to the dosage beyond which any addition of superplasticizer does not change the flow time. In Fig. 6 it is shown the flow time as function of superplasticizer and water/binder ratio, from which it can be observed that the flow time exhibits a trend to decrease with the increase of superplasticizer dosage. In the samples with water/binder of 0.55 the flow time remains almost unchanged which appears to correspond to the maximum degree of dispersion of the grout particles. Moreover, flow time is also reduced with the increase of water/binder ratio; nevertheless this can lead to a worsening of hardened grout by reducing its mechanical properties.

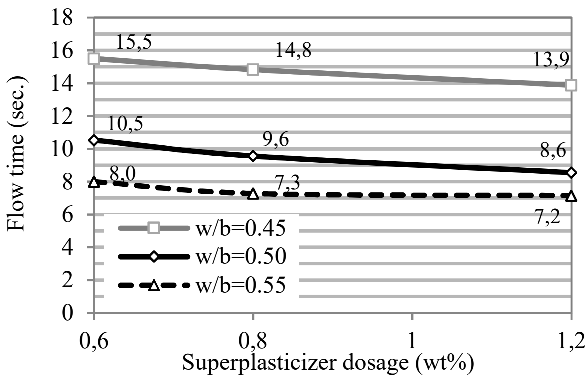

Figure 6. Influence of water/binder ratio and superplasticizer dosage on flow time

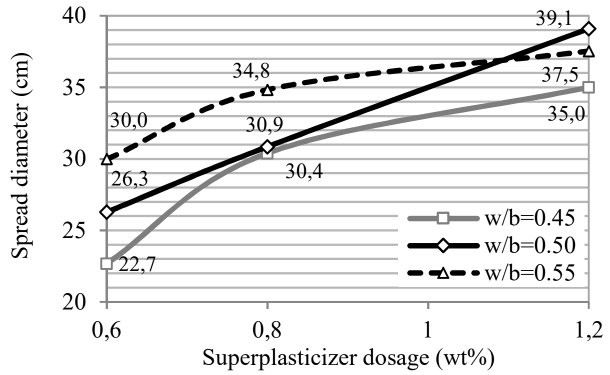

Figure 7. Influence of water/binder ratio and superplasticizer dosage on spread diameter

The results from mini-slump test are shown in Fig. 7. This test gives the spread diameter which is associated with the yield stress, since the grout spread occurs or stops when the shear stress becomes higher or lower than the yield stress. In Fig. 7 it can be observed that grout spread diameter increases with increasing superplasticizer dosage, which is in agreement with the obtained results of yield stress. However, it should be noted that for a superplasticizer dosage of 1.2% the spread diameter does not follow the trend of increase with increasing the water/binder ratio from 0.50 to 0.55. It is belied that this is caused by high superplasticizer concentration; according to some authors [16,17] a higher superplasticizer concentraction will lead to a reverse effect. This phenomenon is called depletion attraction, which is caused by an excessive concentration of superplasticizer in the liquid phase that will be the source of an osmotic pressure over the binder particles forcing the particles to flocculate and causing negative effects such as segregation and bleeding.

Correlation between flow tests and rheological properties. The relationship between the flow time and plastic viscosity is presented in Fig. 8 showing a good correlation (coefficient of correlation R^2 of 0.82). The figure shows that when the flow time increases the same apply to the plastic viscosity. Similarly, the coefficient of correlation between spread diameter and yield stress was also good ($R^2 = 0.87$), as shown in Fig. 9. It can be noted that the increase in spread diameter leads to reduced yield stress values, as expected. The relationship between these properties show that these simple test methods (such as Marsh cone and mini-slump) can be used to predict the plastic viscosity and yield stress value for grout optimization or fresh quality control in situ.

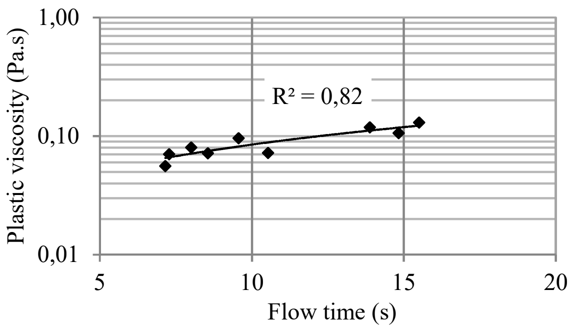

Figure 8. Correlation between plastic viscosity and flow time

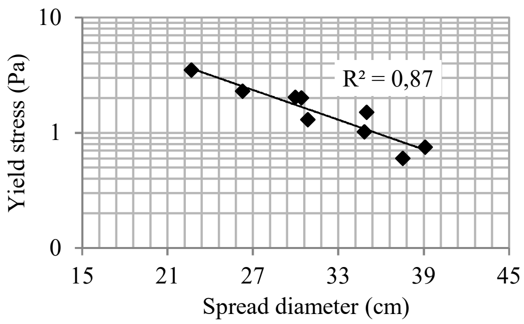

Figure 9. Correlation between yield stress and spread diameter

Mathematical models. As mentioned above, mathematical models were created which allow estimating the grout parameters, such as plastic viscosity and yield stress. Based on the experimental results the models coefficients were calculated using multiple regression analysis. This statistical modelling allows to estimate the value of a dependent parameter, such as yield stress or plastic viscosity, based on a set of independent variables, such as water/binder ratio, superplasticizer dosage, flow time and spread diameter. The general form of the equation of multiple linear regression can be written as follows (Eq. 2):

$$y_1 = \beta_0 + \beta_1 x_{i,1} + \cdots + \beta_{p-1} x_{i,p-1} + \varepsilon_i, i = 1,2, \cdots n, \tag{2}$$

Where y_l means the dependent variable, x_i is the independent variables, β_p is the regression coefficients and ε_i is the additive error. The matrix formulation involving predictor variables $x_1, \cdots, x_p$ takes the following structure (Eq. 3):

$$\begin{pmatrix} y_1 \\ y_2 \\ \cdots \\ y_n \end{pmatrix} = \begin{pmatrix} 1 & x_{1,1} & x_{2,1} & \ldots & x_{p,1} \\ 1 & x_{1,2} & x_{2,2} & \ldots & x_{p,2} \\ & & \ldots & & \\ 1 & x_{1,n} & x_{2,n} & \ldots & x_{p,n} \end{pmatrix} \times \begin{pmatrix} \beta_1 \\ \beta_2 \\ \ldots \\ \beta_p \end{pmatrix} + \begin{pmatrix} \varepsilon_1 \\ \varepsilon_2 \\ \ldots \\ \varepsilon_n \end{pmatrix} \tag{3}$$

The least-squares approach was used to obtain the coefficients of the regression equations. This means, that based on experimental data the regression coefficients $\hat{\beta}$ were calculated in order to minimize the mean square error between the observed and estimated values of y-parameter. That is, to find the regression coefficient $\hat{\beta}$ that minimizes the following criterion [18]:

$$D(\beta) = \sum_{i=1}^{n} (y_i - x_i \beta)^2 \tag{4}$$

Taking derivates with respect to β, and setting these to 0, it is obtained the normal equations:

$$\frac{dD}{d\beta} = -2X'(y - X\beta) = 0 \Rightarrow (X'X)\beta = X'y \tag{5}$$

where X and y are the matrix of variables $x_1, \cdots, x_p$ and $y_1, \cdots, y_n$, respectively. To solve for β the inverse of $X'X$ was applied to both sides of Eq. 5 and the Eq. 6 was obtained:

$$\hat{\beta} = (X'X)^{-1} X'y \tag{6}$$

where $\hat{\beta}$ is the matrix of coefficients.

Proposed models. The least-square approach was used to find the coefficients of each parameter modelled. Moreover, the models give an indication of the relative significance of various variables on each estimated parameter. The majority of the statistical models developed in this study have high coefficients of determination (R^2) of more than 0.80 with the results from experimental work. The models for rheological properties i.e. plastic viscosity and yield stress of the NHL grouts are given in the Eq. (7) and (8), respectively:

$$\text{Plastic viscosity (Pa.s)} = 0.033\ \mathrm{w/b} - 0.043\ \mathrm{SP} + 0.009\ \text{Flow time} + 0.027 \tag{7}$$

$$\text{Yield stress (Pa)} = -6.419\ \mathrm{w/b} - 0.119\ \mathrm{SP} - 0.126\ \text{spread diameter} + 9.041 \tag{8}$$

where *w/b* is the water/binder ratio (-), *SP* is the superplasticizer dosage (wt%) and flow time (sec.) and spread diameter (cm) are the results obtained from experimental work i.e. Marsh cone and mini-cone test, respectively.

Accuracy of the proposed models. The accuracy of the developed models was determined by comparing predicted with measured values obtained from random grout compositions. The results of yield stress and plastic viscosity were then used to verify the ability of the proposed models to predict these parameters. All tests were carried out with under the same conditions and procedures. The comparison between measured and predicted parameters is plotted on Fig. 10 and 11. The high regression coefficient indicates a good agreement between the confirmation results and the expected results from the models. These results indicate that both models together with simple test methods can be used in the prediction as well as in the optimization of fresh performance of NHL grouts.

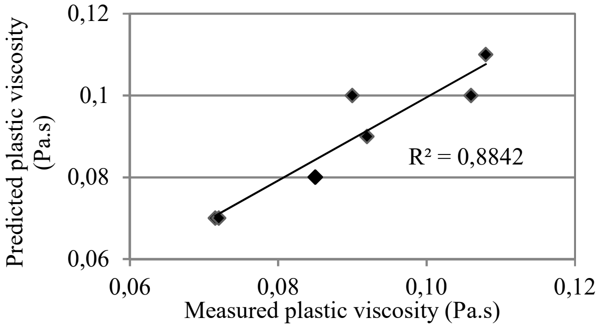

Figure 10. Comparison between the measured plastic viscosity (rheometer) and the plastic viscosity predicted using Eq (7)

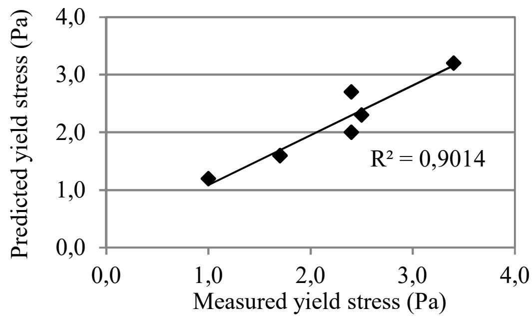

Figure 11. Comparison between the measured yield stress (rheometer) and the predicted yield stress using Eq (8)

Conclusions

This paper has addressed the modelling of the effects of two key components (such as water/binder ratio and dosage of superplasticizer) on the rheological properties of NHL grouts. The influence of these key components on rheological properties, flow time and spread diameter was analysed. The good correlation between the flow tests (i.e. Marsh cone test and mini-slump test) and the rheological parameters (yield stress and plastic viscosity) indicated that these simple flow tests could be used to predict the plastic viscosity and yield stress. Hence, the grout components together with the outputs from the flow tests were used for the development of mathematical models. These models can be very useful to evaluate and to adjust the grout composition in order to achieve the fresh properties that suit each particular masonry requirements. Based on the results obtained the models are valid within the range of design components and provide an efficient mean to determine the influence of theses components on grout rheological properties. It should be noted that the modelling was done based on a given set of materials and assumptions which means that the generalisation of the results should be considered carefully. Therefore, further experimental research is needed to know how the differences in the properties between the present NHL and other NHLs might affect the modelling results. The difference between the predicted and measured values will indicate the effect of the new ranges on the accuracy of the proposed models. In any case, the proposed models allow simplifying the whole methodology involved in grout design by reducing time and resources involved in rheological measurements, especially when complex equipment such as rheometer or viscometer is involved. The results summarised in this paper are part of a research program of the civil engineering department of *University Nova of Lisbon* and aim to contribute to the improvement of grouts performance as well as to optimize the procedures associated with injection tests performed into different porous media simulating old masonries.

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