EXPERIMENTAL STUDY AND MODELING OF RHEOLOGICAL AND MECHANICAL PROPERTIES OF NHL GROUTS

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ABSTRACT

This paper aims to model the effect of grout composition on properties of two natural hydraulic lime grouts based on the correlation between grout rheometer results and simple flow tests. Firstly, the effects of water/binder ratio and superplasticizer dosage on its rheological properties and flowability were analyzed. Dosage of superplasticizer and water/binder ratio were varied from 0.6% to 1.2% (by mass of binder) and 0.45 to 0.55, respectively. A good correlation between classical flow tests and the rheological properties was obtained. Then, statistical models were formulated in order to estimating the grout parameters, such as plastic viscosity and yield stress just by performing simple flow tests. The models coefficients were calculated using multiple regression analysis. The statistical modeling results indicated that the properties of the grouts studied are linearly related to water/binder ratio, superplasticizer dosage and specific surface area of natural hydraulic limes. Finally, the accuracy of the models was experimentally confirmed using random grout compositions. The predicted-to-measured ratio ranged from 0.97 to 1.08, indicating a good agreement between the confirmation results and the expected results from the statistical models.

Keywords: *Grout, natural hydraulic limes, superplasticizer, Marsh cone test, mini-slump test, rheology*

INTRODUCTION

Multi-leaf stone masonry walls are a masonry typology with particular weaknesses, namely weak monolithic behavior under both horizontal and vertical loads. Due to the presence of poor materials in the inner core, voids and scarce of connections between elements this masonry typology is prone to detach from the rest of the building and to fail out-of-the plane by disaggregating into different parts (Binda et al 2006; Mauro and Felice 2012). Grout injection is an interesting consolidation technique to repair and reinforce such walls. This method 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 so that the load on the wall is distributed evenly over the whole bearing area of the masonry, and bond the delaminated layers together and help them resist lateral forces.

Grouts are mixtures of a binder with water and admixtures, which must feature adequate fluidity and penetrability in order to be injectable. However, choosing appropriated grout is often a critical phase. In most cases, the selection of grout depends on the desired fresh properties and performance characteristics. It has been pointed out that fluidity and injection capacity are essential fresh properties (Jorne et al. 2014; Baltazar et al. 2014; Baltazar et al. 2012). The control of fluidity can be done through rheological measurements. Currently, rheology is often used as a tool in the control and design of cementitious suspensions, such as cement based pastes, mortars, concretes and grouts (Cardoso et al. 2014; Baltazar et al. 2013; Roussel et al. 2010; Roussel 2007). Therefore, rheological measurements using rheometers or viscometers can be expensive and complex to operate, and also give too many information when often just two rheological parameters are needed, such as yield stress and plastic viscosity (Baltazar et al. 2013; Baltazar et al. 2015). In this framework, this paper aims to model the effect of water/binder ratio and dosage of superplasticizer on the rheological properties of natural hydraulic lime (NHL) grouts using simple tests easy to use at construction sites, such as Marsh cone and spread test using a mini-slump flow. For this reason the statistical modeling approach was used in order to establish models for predicting 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. Additionally, two more models are proposed to help predicting the compressive and flexural strength of those grouts.

This paper is part of a larger study and aims to help to achieve an adequate balance between the grout rheological properties and its injectability, which can be tested in reduced models simulating old masonries as the type used in (Jorne et al. 2014). Only then, based on the injection tests, can the composition of those grouts be tailored according to the properties of the masonry to which such a grout is to be injected. In practice, the variability of voids within masonries requires an ability to fine tune the rheological properties of the grout in order to get a successful consolidation process. The methodology presented in this research will provide necessary tools for that purpose.

METHODOLOGY

The influence and significance of each grout design variable on its rheological behavior was identified in a previous work using the design of experiments method (DOE) together with analysis of variance (ANOVA) (Baltazar et al. 2013). Based on these previous results, the water/binder ratio and dosage of superplasticizer were pointed out as the most significant grout components. The first part of this paper will focus on the effects of these two grout components on yield stress, plastic viscosity, flow time, spread diameter and mechanical strength. In the second part, a correlation between rheological properties and the flow time and spread diameter results is made, in order to formulate statistical models for prediction of the rheological properties associated to each grout. Finally, in a third part, the accuracy of the models is experimentally confirmed using random grout compositions.

STATISTICAL MODELS

Statistical models were created for estimating the grout parameters, such as plastic viscosity and yield stress. This statistical modeling 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, specific surface area of NHL used and spread diameter. The general form of the equation of multiple linear regression can be written as follows (eq. 1):

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

Where y_1 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. 2):

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

The first step in multiple linear regression analysis is to determine the matrix of coefficients ($\hat{\beta}$). 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 (Anderson 1984):

$$D(\beta) = \sum_{i=1}^{n}(y_i - x_i\beta)^2 \quad (3)$$

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

$$\frac{dD}{d\beta} = -2X'(y - X\beta) = 0 \Rightarrow (X'X)\beta = X'y \quad (4)$$

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. 4 and the eq. 5 was obtained:

$$\hat{\beta} = (X'X)^{-1}X'y \quad (5)$$

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

EXPERIMENTAL DETAILS

Materials

The experimental program was conducted using two types of NHL, namely NHL3.5 and NHL5 produced according to the European Standard EN459-1:2010. NHL was chosen as binder, since it is a hydraulic binder that presents chemical and physical properties closer to those of pre-existing materials in old masonries and is able to set both in dry and wet conditions, with or without contact with air (Baglioni et al. 1997; Binda et al. 2006).The physical and chemical properties of both NHLs are listed in table 1. The grain size distribution was determined in dry powder samples using a laser diffraction particle size analyzer (Coulter LS 230), the results are represented in Fig. 1. A comparison of these two NHL types revealed that NHL3.5 had a larger specific surface area than NHL5. A commercially available polycarboxylate ether superplasticizer (i.e. high range water reducer) conforming to ASTM C494-05 Type F was used. It had a specific gravity, pH, chloride content, charge and solid content of 1.05, 8, <0.10%, anionic and 28-32%, respectively.

Mixing procedure

All grouts were prepared in laboratory in batches of 4 liters, and mixed using a high shear mixer equipped with helicoidal blade (see Fig. 2). For the preparation of grouts ordinary tap water was used. The mixing procedure adopted was obtained in previous research using the design of experiments method (Baltazar et al. 2012): the whole lime is added to 70% of total mix water (the water was first introduced in the mixer and then the binder) and mixed for 10 minutes. The remaining water (with diluted superplasticizer) is added within 30s. After all materials had been added, the mixture was maintained for an additional 3 minutes. The mixing speed during the whole mixing was 800 rpm. The hydraulic lime grouts were prepared at ambient temperature of 20±2ºC and a relative humidity of 60±5%. Following the end of mixing, the grout flow tests as well as the rheological measurements were performed as described below.

Experimental procedures

As mentioned, in this study two key grout design variables were chosen to use in formulating models for evaluating fresh and hardened properties. The values of each design variable are summarizes in table 2. In total, eighteen grout combinations were tested (nine for each type of NHL). The ranges of water/binder ratio and superplasticizer dosage were proportioned so that the grouts exhibit acceptable performance to be used in masonry injection. Therefore, the proposed models have a valid domain for grouts made with 100% of NHL, water/binder ratio of 0.45 – 0.55 and superplasticizer dosage range of 0.6 – 1.2%, by mass of lime. The ranges of water/binder ratio and superplasticizer dosage were proportioned so that the grouts exhibit acceptable performance to be used in masonry injection. Water/binder ratio lower than 0.45 results in grouts with insufficient workability and therefore impossible to be injected (Miltiadou, 1990). On the other hand, water/binder ratio higher than 0.55 leads to an excessive bleeding of free water that compromises the grout stability and therefore its homogeneity until onset of hardening. 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 composition (see table 2) a total of 3200g were used in each batch of preparation, 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.

Rheological measurements

A rotational rheometer was used to evaluate rheological properties of NHL grouts, including yield stress and plastic viscosity. The geometry used consisted of a plate-plate geometry (with Ø = 40 mm) and a gap of 2 mm (see Fig. 3). In all measurements the testing protocol consisted on subjecting the grout to a pre-shearing stage of 60s at shear rate of $1s^{-1}$ followed by 60s at rest. 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 $300s^{-1}$ (the maximum shear rate used – see Fig. 4). 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 analyzed 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. 6) was adopted to describe the grout rheological behavior and to determine the yield stress and plastic viscosity of fresh grouts (Barnes 2000):

$$\tau = \tau_0 + \eta_p \times \dot{\gamma} \qquad (6)$$

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

Marsh cone test

The Marsh cone test was performed according to ASTM C939-02. Based on this standard the measurement of flow time is connected to the grout fluidity. In order to improve the physical significance of Marsh cone test (especially for grouts with high water/binder ratio), the grout fluidity was evaluated using a modified Marsh cone having an outlet diameter of 5 mm. A volume of 1,000 ml of grout was placed into the cone and the flow time (expressed in seconds) refers to the time required for 800 ml of grout to flow through the cone.

Mini-slump test

Besides the Marsh cone test, the slump flow was also performed. In this work the mini-slump test adopted is similar to the procedure developed by Roussel in (Roussel et al. 2005), in order to try to correlate it with the yield stress measured in the rheometer. The mini-cone used has an height of 50mm and a top and bottom diameter equal to 35mm and 50mm, respectively.

Mechanical Strength

In order to determine the mechanical characteristics of the formulated grouts, the flexural and compressive strengths were evaluated with five samples of each grout, which were poured into steel moulds (160x40x40 mm). After 5 days, the specimens were taken out from the moulds and cured in a controlled atmosphere at 20±2ºC and 60±5% relative humidity until the age of maturity of 28 days. The load was applied at constant rate of 0.5mm/min and 0.7mm/min for flexural and compressive strength, respectively using a Z050 Zwick mechanical test machine with 50 kN capacity following standard EN 1015-11:1999. In order to understand the previous results, the evaluation of entrained air in fresh grout was measured based on the standard EN 492-2:2002. Moreover, the effect of superplasticizer on the grout microstructure was carried out using a Zeiss DSM 962 Scanning Electron Microscope (SEM) at an accelerating voltage of 5kV.

RESULTS AND DISCUSSION

Rheological measurements

Typical shear stress *vs*. shear rate profile determined for the grout with water/binder of 0.50 and superplasticizer dosage of 0.8% is shown in Fig. 5 (the viscosity profile is shown in Fig. 6). The profiles obtained for the other grouts are qualitatively similar. The relationship between shear stress and shear rate shows that all grouts (both types of NHL) have shear-thinning behavior. However, it should be pointed out that, the high shear-thinning observed may be caused by some slippage at the plate-material interface that can occur at low shear rates, since smooth plates were used (Barnes, 1997). Notwithstanding, it is believed that the shear-thinning behavior also occurred owing to the elongated shape of NHL particles (Toumbakari, 2002; Bras, 2011). Moreover, and according to some authors (Banfill 2006; Saak et al, 2001; Nehdi and Rahman, 2004) the slippage can also contribute to underestimation of yield stress values. Therefore, in order to mitigate the effect of eventual slippage, the yield stress values were determined as the intercept of eq 6 at zero shear rate (as shown in Fig 5).

The effect of superplasticizer dosage and water/binder ratio on grout rheological properties is presented in Fig. 7 and 8. It is known that superplasticizers enable to impose repulsive forces that prevent the particle flocculation. Comparing the two types of NHL it is clear that (at the same composition) the NHL3.5 has higher yield stress and plastic viscosity values than NHL5, which is due to higher specific surface of NHL3.5 that makes less superplasticizer and water available to promote fluidity. Consequently, the internal friction among solid particles increases, leading to higher yield stress and plastic viscosity. Moreover, regardless the type of NHL it can be seen that the yield stress is more affected with increasing superplasticizer dosage than the plastic viscosity. As expected, the water/binder ratio has a similar behavior to the superplasticizer dosage, however, simple addition of water to make the grout more fluid is not an efficient solution because more water also increases porosity and consequently reduces mechanical properties.

Marsh test and mini-slump test

The Marsh cone test is a simple test which can be used to characterize the workability of fresh grout in the laboratory and in the field. The flow time seems to be a relevant parameter for estimating the grout plastic viscosity. The Marsh cone value as function of superplasticizer dosage and water/binder ratio is shown in Fig. 9, from which it can be observed that the flow time is influenced by both superplasticizer dosage and water/binder ratio and it exhibits a trend to decrease with their increase. The range of change observed in flow time between the two types of NHL depends on the NHL specific surface area, which corroborates the rheological parameters previous determined.

From the results of mini-slump test presented in Fig. 10 it can be concluded that grout spread diameter increases with increasing superplasticizer dosage, which is in agreement with the obtained results of yield stress. However, care must be taken when using high dosages of superplasticizer. One can see in Fig. 10 that for NHL5 grout at the superplasticizer dosage of 1.2% the spread diameter is lower for water/binder ratio of 0.55 than for 0.50. It is believed that this behavior is due to the joint effect of high water/binder ratio with excessive unabsorbed superplasticizer present in the suspension. According to some authors (Banfill 2011; Flatt 1999) a higher superplasticizer concentraction lead to a reverse effect. Regarding the NHL3.5 this behavior was not observed as result of its high surface area.

Correlation between flow tests and rheological properties

The relationship between the flow time and plastic viscosity is presented in Fig. 11 showing a good correlation (coefficient of correlation R^2 of 0.82 and 0.79 for NHL5 and NHL3.5, respectively). 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 of 0.71 and 0.92 for NHL5 and NHL3.5, respectively), as shown in Fig. 12. It can be noted that the increase in spread diameter leads to reduced yield stress values, as expected. The relationship between these properties shows 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.

Mechanical strength

The grout mechanical strength is of great importance since the behavior of hardened grout has a determinant effect on the mechanical properties of the grouted masonry. The average measurements of flexural and compressive strength of NHL5 and NHL3.5 grouts at the maturity age of 28 days with different water/binder ratios and superplasticizer dosages are shown in Fig. 13 and 14. The obtained results and their analysis indicate that the general tendency is that mechanical strength increases up to a superplasticizer dosage of 0.8 wt%, which means that superplasticizer can be useful for improvement of mechanical properties.

The NHL hydration process and mechanism of the superplasticizer effects are out of the scope of this study. However, representative samples of grouts with and without superplasticizer were examined using a scanning electron microscope (SEM). Grout sample, containing superplasticizer, observation shows that instead of larger and well-defined crystals, smaller crystals and denser microstructure are formed (see Fig.15). Notwithstanding, a slight downward trend in the mechanical strength values between 0.8 and 1.2 wt% has been observed. According with the results of air entrained presented in Fig 16 it can be concluded that the presence of high concentration of superplasticizer leads to a slight increase of air incorporated in the fresh grouts, which may also explain the reduction in mechanical strength.

Proposed models

Based on the experimental results the least-square approach was used to found the coefficients of each modeled parameter. In the following steps the least-square approach is illustrated. The first step was the product of X' and X as follows:

$$A = X'.X$$

where *X* is the matrix of independent variables

$$A = \begin{bmatrix} 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 & 1 \\ .55 & .55 & .55 & .50 & .50 & .50 & .45 & .45 & .45 & .55 & .55 & .55 & .50 & .50 & .50 & .45 & .45 & .45 \\ .6 & .8 & 1.2 & .6 & .8 & 1.2 & .6 & .8 & 1.2 & .6 & .8 & 1.2 & .6 & .8 & 1.2 & .6 & .8 & 1.2 \\ 598 & 598 & 598 & 598 & 598 & 598 & 598 & 598 & 598 & 480 & 480 & 480 & 480 & 480 & 480 & 480 & 480 & 480 \end{bmatrix} \times \begin{bmatrix} 1 & .55 & .6 & 598 \\ 1 & .55 & .8 & 598 \\ 1 & .55 & 1.2 & 598 \\ 1 & .50 & .6 & 598 \\ 1 & .50 & .8 & 598 \\ 1 & .50 & 1.2 & 598 \\ 1 & .45 & .6 & 598 \\ 1 & .45 & .8 & 598 \\ 1 & .45 & 1.2 & 598 \\ 1 & .55 & .6 & 480 \\ 1 & .55 & .8 & 480 \\ 1 & .55 & 1.2 & 480 \\ 1 & .50 & .6 & 480 \\ 1 & .50 & .8 & 480 \\ 1 & .50 & 1.2 & 480 \\ 1 & .45 & .6 & 480 \\ 1 & .45 & .8 & 480 \\ 1 & .45 & 1.2 & 480 \end{bmatrix}$$

$$= \begin{bmatrix} 18 & 9 & 15.6 & 9702 \\ 9 & 4.53 & 7.8 & 4851 \\ 15.6 & 7.8 & 14.64 & 8408.4 \\ 9702 & 4851 & 8408.4 & 5292036 \end{bmatrix}$$

In the next step the inverse matrix was determined:

$$K = (A)^{-1} = \begin{bmatrix} 13.696 & -16.667 & -0.774 & -0.01 \\ -16.667 & 33.333 & -7.9E-14 & -9.58E-16 \\ -0.774 & -6.5E-14 & 0.893 & 5.227E-18 \\ -0.001 & -1E-15 & 2.42E-30 & 1.596E-5 \end{bmatrix}$$

Using this matrix *K*, it can now be determined the coefficients for each model:

$$\hat{\beta} = K.X'.y$$

where *y* is the matrix of the outputs from each experimental test, such as the spread diameter, flow time, etc. in order to determine the coefficient of the corresponding regression model. The variables coefficients and their contribution for the modeled parameters are present in table 3. The presentation in table 3 gives 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 0.86 with the results from experimental work. The models for rheological properties (plastic viscosity, yield stress) and mechanical strength (flexural and compressive strength) of the NHL grouts are given in the eqs (7), (8) and eqs. (9), (10), respectively:

$$\text{Plastic viscosity (Pa.s)} = 0.119\,\text{w/b} + 0.035\ \text{SP} + 0.015\ \text{flow time} + 0.001\ \text{SS} - 0.390 \quad (7)$$

$$\text{Yield stress (Pa)} = -113.362\,\text{w/b} + 12.586\ \text{SP} - 3.324\ \text{spread diameter} - 0.041\ \text{SS} + 189.386 \quad (8)$$

$$\text{Flexural strength (MPa)} = -4.592\,\text{w/b} - 0.419\ \text{SP} - 0.001\ \text{SS} + 5.167 \quad (9)$$

$$\text{Compressive strength (MPa)} = -17.560\,\text{w/b} - 1.152\ \text{SP} - 0.01\ \text{SS} + 20.765 \quad (10)$$

where *w/b* is the water/binder ratio, *SP* is the superplasticizer dosage (%), *SS* is the specific surface area of NHL (m^2/kg) and flow time (sec.) and spread diameter (cm) are the results obtained from experimental work i.e. Marsh cone and mini-cone test, respectively.

Effects of water/binder ratio and superplasticizer dosage on plastic viscosity and yield stress

The proposed models can therefore be used to create contour diagrams showing the influence of components on the properties that affect the fresh performance of NHL grouts. As shown in eq. 7, the water/binder ratio has a higher effect than superplasticizer dosage over plastic viscosity. Fig. 17 shows the effect of varying the superplasticizer dosage and flow time on the grout plastic viscosity with water/binder ratio of 0.50. Graphically the contour lines show the isoresponse of plastic viscosity when the superplasticizer dosage and flow time (obtained from Marsh cone test) are known. For instance, as can be seen in Fig. 17, a grout made with 0.50 of water/binder ratio and 1% of superplasticizer can exhibit an increase in plastic viscosity from 0.34 Pa.s to 0.48 Pa.s when the flow time increased from 10s to 20s. Note, however, that this isoresponse plot is only valid for water/binder ratio of 0.50 and NHL5. As far as yield stress is concerned, it is more significantly affected by the water/binder ratio than the superplasticizer dosage as shown in eq. 8. The effect of varying the superplasticizer dosage and spread diameter on yield stress values at water/binder ratio of 0.50 is shown in Fig. 18. Assuming, for instance, a grout mix with a superplasticizer dosage of 0.8 % and a spread diameter of 25 cm it is expected that the yield stress will have a value around 40.0 Pa.

Effect of water/binder ratio and superplasticizer dosage on flexural and compressive strength

The effects of water/binder ratio and superplasticizer dosage on the grout mechanical strength at 28 days are shown in eqs. 9 and 10. The water/binder ratio affects the flexural and compressive strengths more significantly than the superplasticizer dosage. The effect of grout components on the compressive and flexural strength are presented in Fig 19 and 20, respectively. However, a trade-off between water/binder ratio and superplasticizer dosage must be done. Since, higher dosages of water (w/b>0.50) have a harmful effect on the hardened grout properties as result of excessive mixing water. Moreover, it can be noticed that higher superplasticizer dosages also reduce the grout mechanical strength, confirming the negative effect of high superplasticizer dosage observed by other authors (Agull et al. 1999; Banfill 2011; Hallal et al. 2010). Notwithstanding, the authors believe that a grout should have high injectability and adequate mechanical properties, the latter are less determinant than the former to get a successful grouting since the typical compressive stresses in old masonry in the range of 1 MPa and most of the analyzed grout compositions are able to conform with these values.

Accuracy of the proposed models

The accuracy of the developed models was determined by comparing predicted-to-measured values obtained from ten random grout compositions (see table 4). The results of yield stress, plastic viscosity, compressive and flexural strengths were then used to verify the ability of the proposed models to predict these parameters. All tests were carried out under the same conditions and procedures described earlier.

The maximum, minimum, average, standard deviation (SD) and coefficient of variation (COV) of the predicted-to-measured values for yield stress, plastic viscosity, compressive and flexural strengths for all the grout compositions tested are presented in table 5. The results reveal that the predicted-to-measured ratio ranged from 0.97 to 1.08, indicating a good agreement between the confirmation results and the expected results from the models. Thus, it can be stated that all the proposed models together with simple test methods can be used in the prediction as well as in the optimization of fresh and hardened performance of NHL grouts.

CONCLUSIONS

This paper provides an approach that simplifies the whole methodology involved in NHL grout design by reducing time and resources involved in rheological measurements, especially when complex equipment is needed, such as rheometer or viscometer, which is not readily available in every field. Concerning the results of this research, the following conclusions can be warranted:

- The statistical modeling results indicate that the plastic viscosity, yield stress, compression and flexural strength of the grouts studied are linearly related to water/binder ratio, superplasticizer dosage and specific surface area of NHLs.
- The predicted-to-measured ratio ranged from 0.97 to 1.08, indicating a good agreement between the confirmation results and the expected results from the models.
- The proposed statistical models together with classical flow tests can provide an efficient mean to determine the grout composition that best fits the requirements of the masonry to which such grouts is to be injected.
- It should be noted that the modeling was done based on a given set of materials and assumptions which means that the generalization of the results should be considered carefully. Therefore, further experimental research is needed to validate these equations using different ranges of test parameters.

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Table 1
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	NHL3.5		NHL5	
Compression resistance at 28 days (MPa)	3.5		5.5	
Setting time	Start	1h	Start	2 h
	End	6h	End	6 h
Specific gravity	2.68 g/cm^3		2.73 g/cm^3	
Specific surface area B.E.T.	598 m^2/kg		480 m^2/kg	
Al_2O_3	2.90		2.00	
CaO	82.00		85.00	
Fe_2O_3	2.04		2.00	
MgO	1.10		1.00	
MnO	0.02		0.03	
SiO_2	8.90		8.00	
SiC	0.03		0.01	
SO_3	1.80		1.00	
SrO	0.06		0.05	
K_2O	0.83		0.70	

Table 2
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Designation	water/binder (g/batch)	superplasticizer (g/batch)
G1	0.45 (1440)	0.6wt% (19.2)
G2	0.45 (1440)	0.8wt% (25.6)
G3	0.45 (1440)	1.2wt% (38.4)
G4	0.50 (1600)	0.6wt% (19.2)
G5	0.50 (1600)	0.8wt% (25.6)
G6	0.50 (1600)	1.2wt% (38.4)
G7	0.55 (1760)	0.6wt% (19.2)
G8	0.55 (1760)	0.8wt% (25.6)
G9	0.55 (1760)	1.2wt% (38.4)

Table 3
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	Plastic viscosity	Yield stress	Flexural strength	Compressive strength
Intercept	-0.390	189.386	5.167	20.765
water/binder	0.119	-113.362	-4.592	-17.560
Superplasticizer	0.035	12.586	-0.419	-1.152
Specific surface area	0.001	-0.041	-0.001	-0.01
Flow time	0.015	-	-	-
Spread diameter	-	-3.324	-	-
R^2	0.96	0.90	0.89	0.86

Table 4

Designation	water/binder	Superplasticizer (wt%)	NHL type
G10	0.45	0.9	NHL3.5
G11	0.46	1.2	NHL3.5
G12	0.48	0.8	NHL3.5
G13	0.48	0.8	NHL5
G14	0.46	1.2	NHL5
G15	0.48	0.9	NHL5
G16	0.52	0.7	NHL3.5
G17	0.52	0.7	NHL5
G18	0.54	0.6	NHL3.5
G19	0.54	0.6	NHL5

Table 5

Property	Predicted-to-measured ratio				
	Maximum	Minimum	Average	SD	COV (%)
Yield stress	1.08	0.92	1.00	0.06	5.60
Plastic viscosity	1.12	0.82	0.97	0.12	12.30
Compressive strength	1.20	0.93	1.08	0.11	9.76
Flexural strength	1.18	0.96	1.06	0.06	6.12
COV=SD/average					

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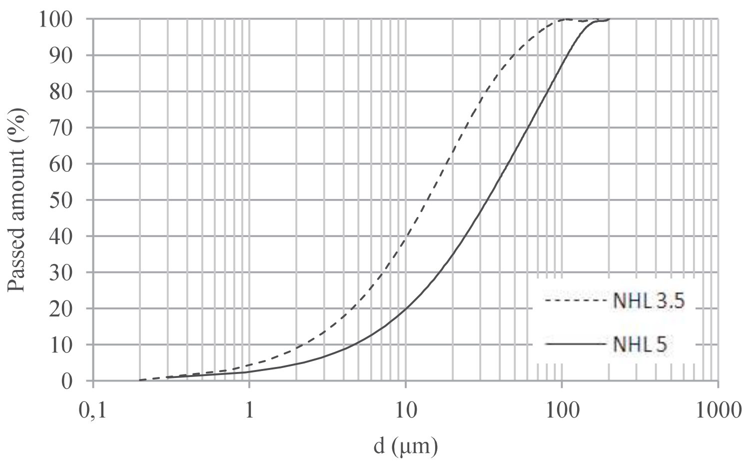

Fig 2
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Fig 3(a)
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Fig 3(b)
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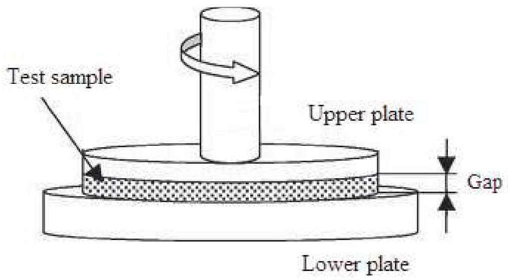

Fig 4

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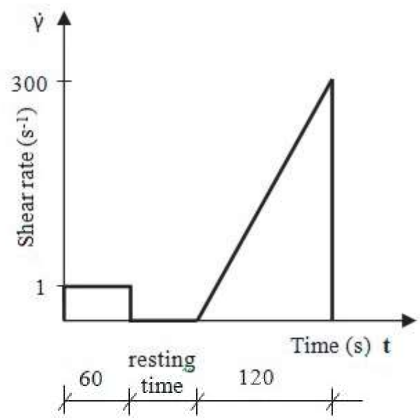

Fig 5

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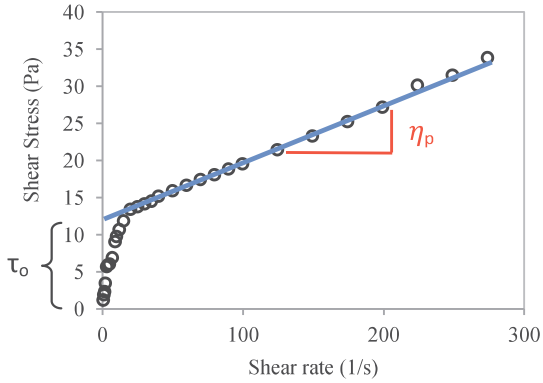

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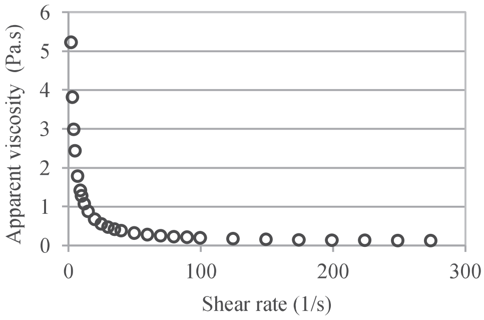

Fig 7

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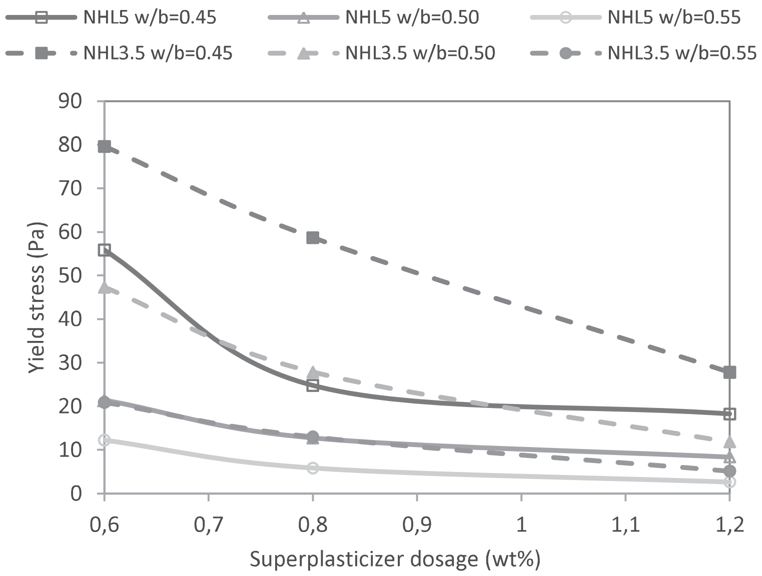

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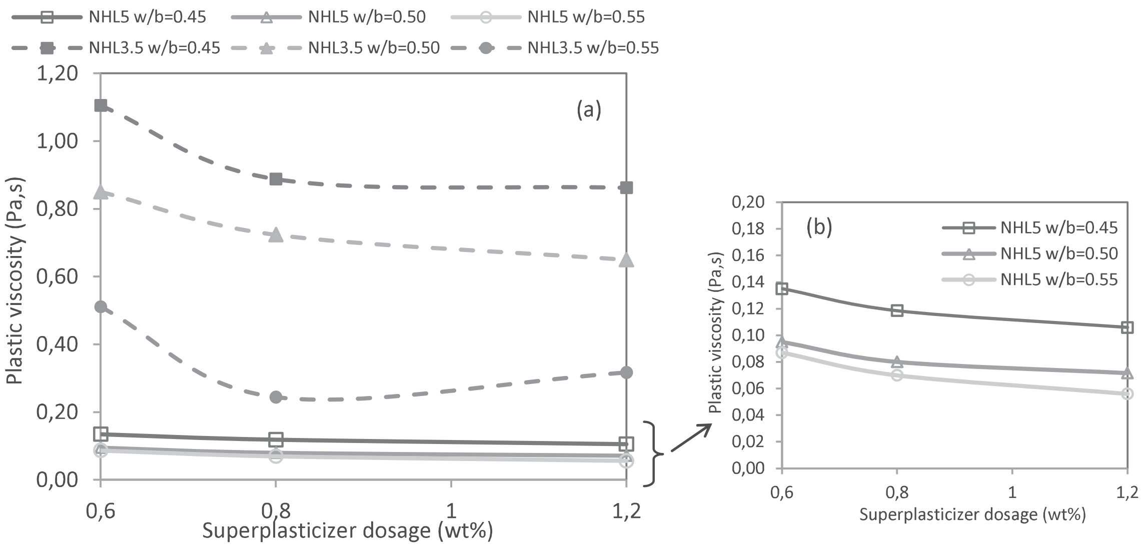

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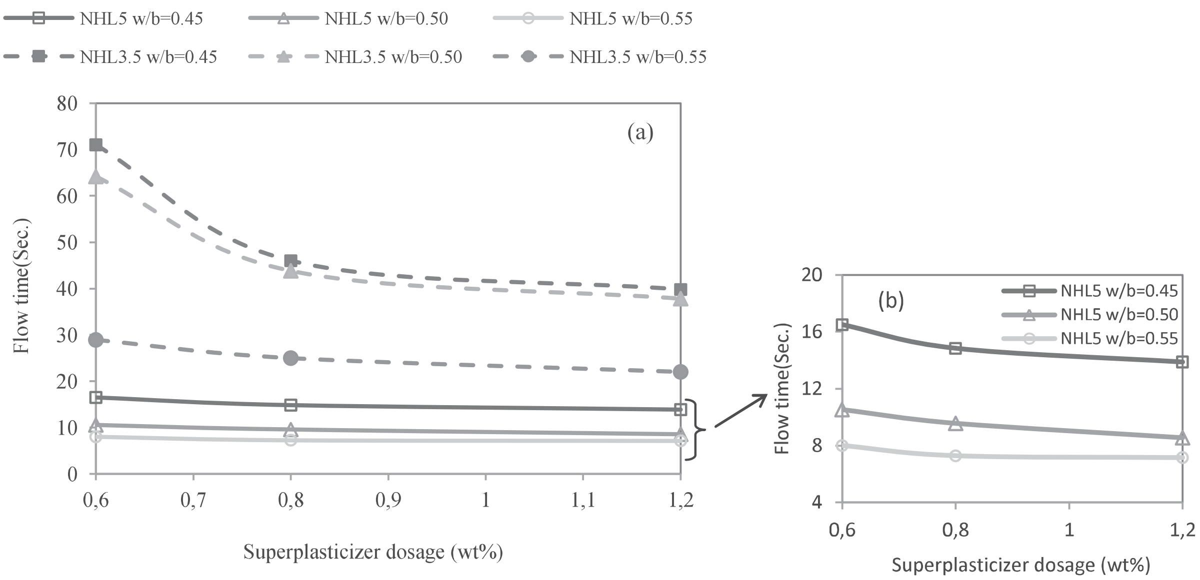

Fig. 10
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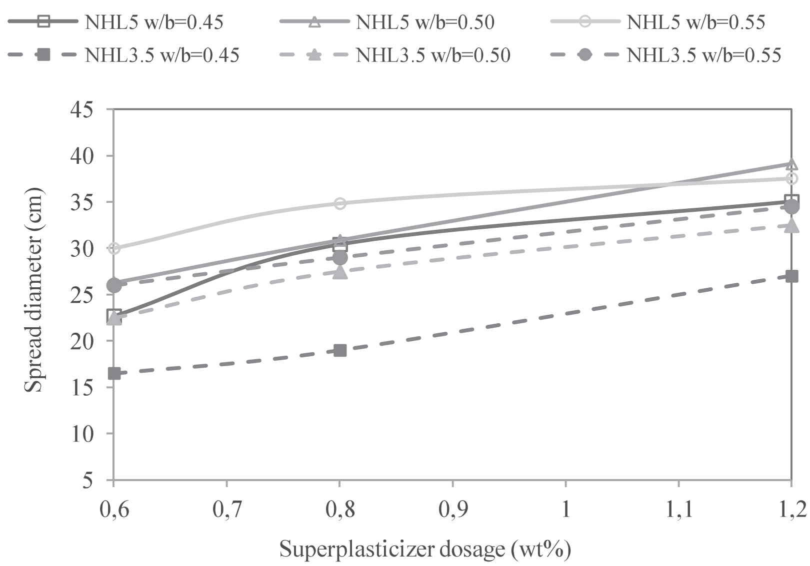

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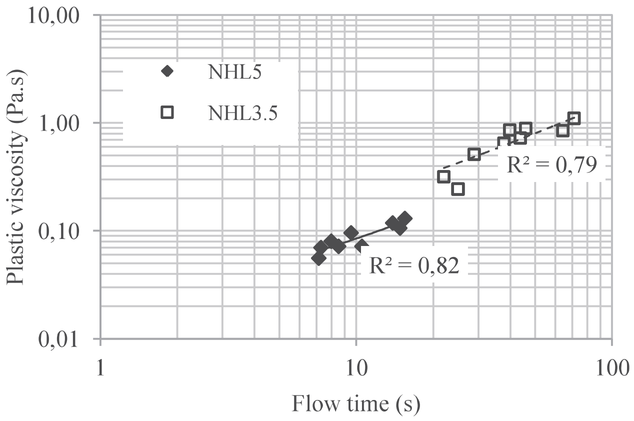

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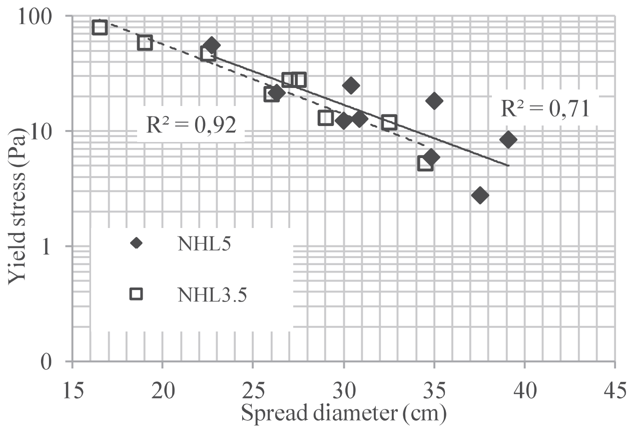

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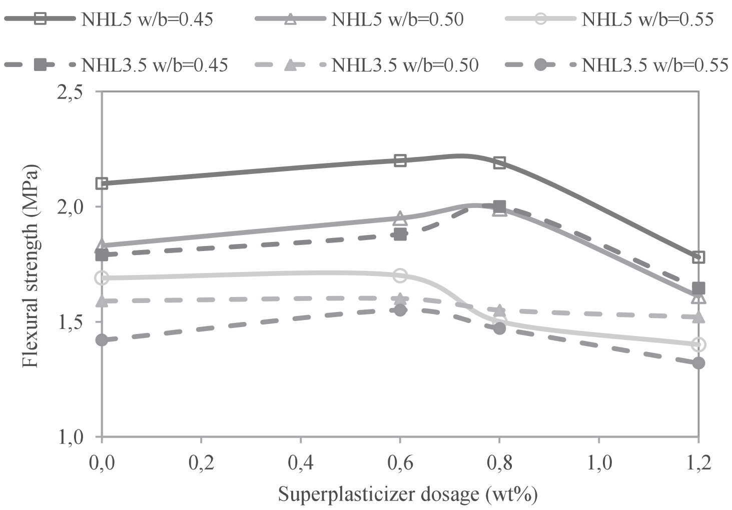

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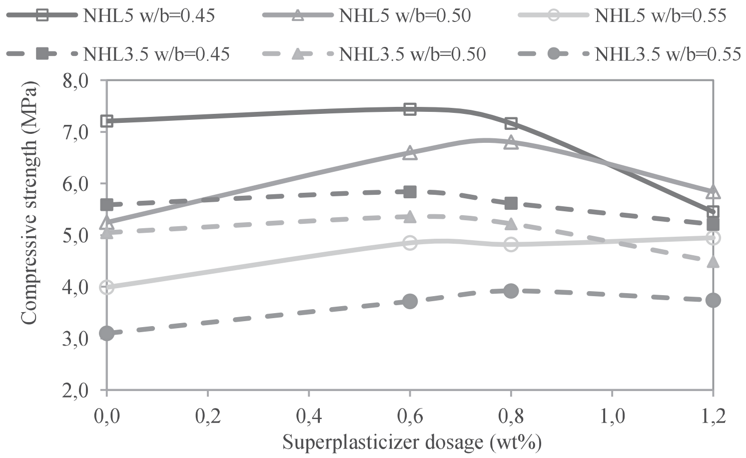

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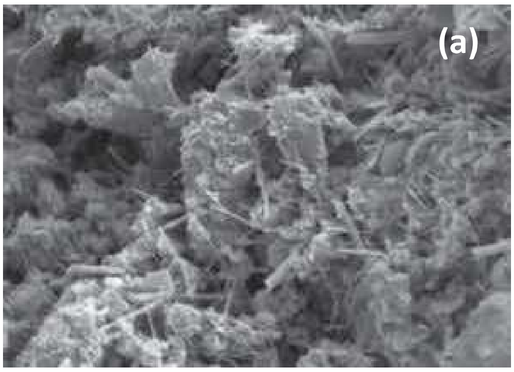

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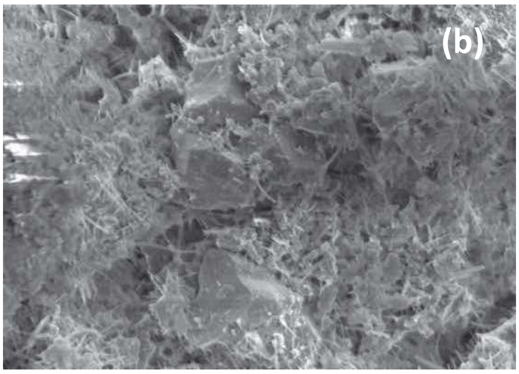

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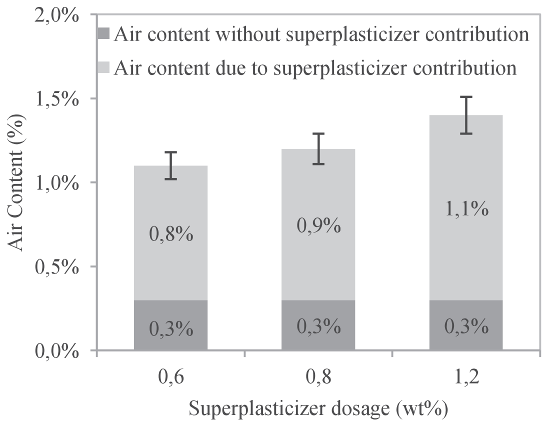

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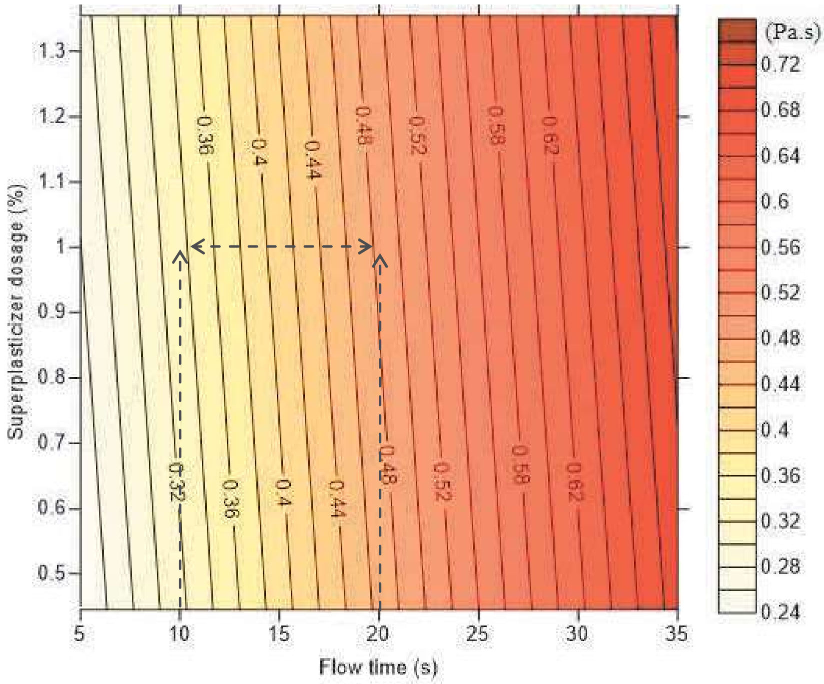

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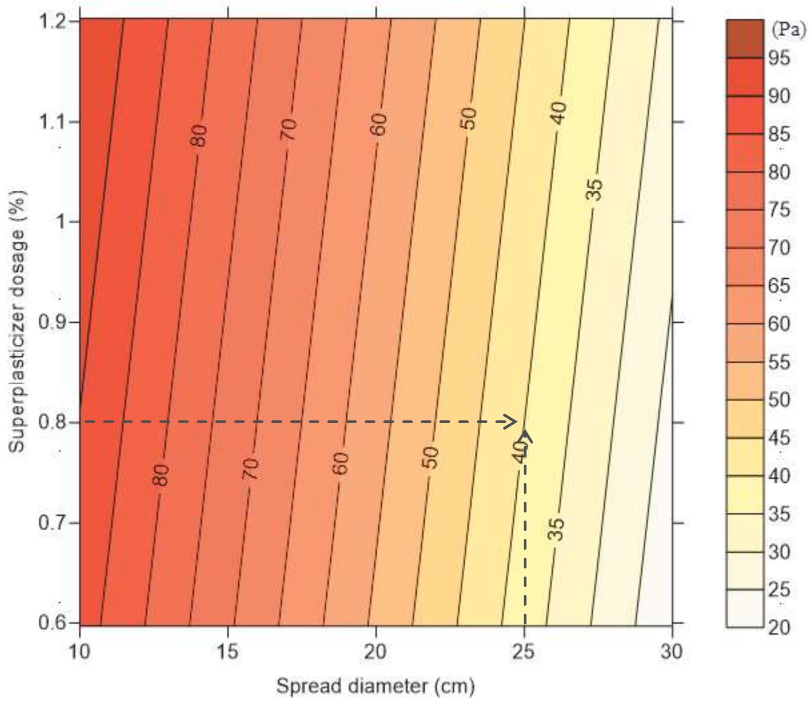

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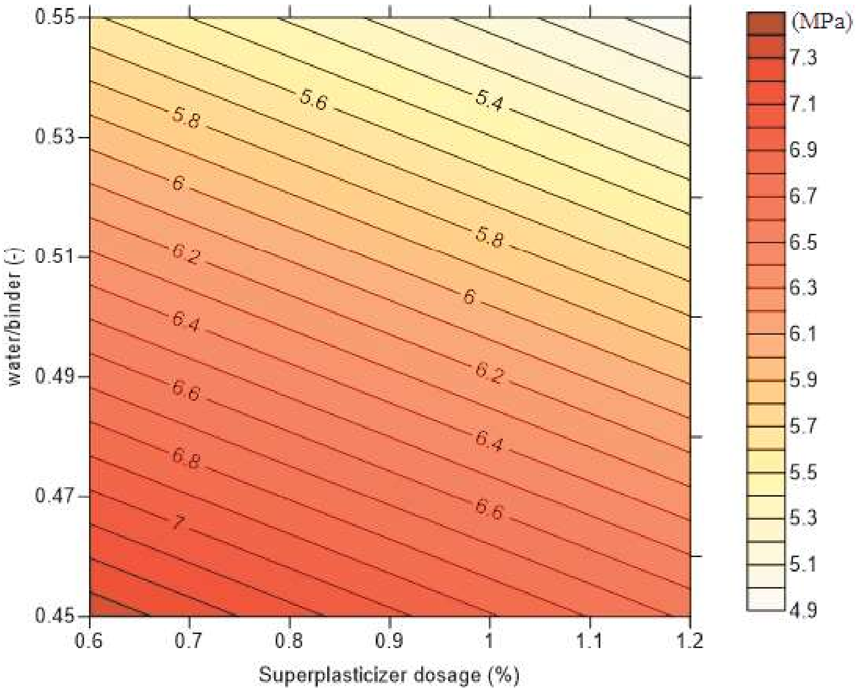

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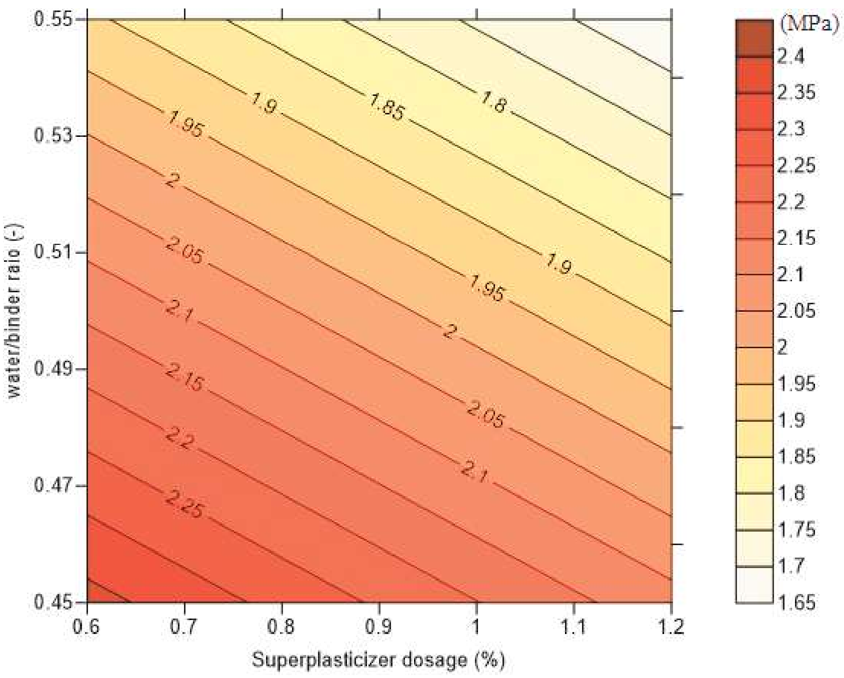

Figure 1 Grain size distribution of both NHL used

Figure 2. Mixer apparatus used in the experimental work

Figure 3. (a) geometry being loaded and (b) plate-plate geometry

Figure 4. Schematic representation of shear rate history adopted for rheological measurements

Figure 5. Shear stress versus shear rate for the NHL 5 grout with w/b=0.50 and SP=0.8%

Figure 6. Apparent viscosity versus shear rate for the NHL 5 grout with w/b=0.50 and SP=0.8%

Figure 7. Influence of water/binder ratio and superplasticizer dosage on yield stress

Figure 8. (a) Influence of water/binder ratio and superplasticizer dosage on plastic viscosity; (b) focus on NHL5 grouts

Figure 9. (a) Influence of water/binder ratio and superplasticizer dosage on flow time. (b) focus on NHL5 grouts

Figure 10. Influence of water/binder ratio and superplasticizer dosage on spread diameter

Figure 11. Correlation between plastic viscosity and flow time

Figure 12. Correlation between yield stress and spread diameter

Figure 13. Influence of water/binder ratio and superplasticizer dosage on grout flexural strength results at 28 days

Figure 14. Influence of water/binder ratio and superplasticizer dosage on grout compressive strength results at 28 days

Figure 15. SEM image of 7 days hardened grout (NHL5 + w/b=050) at 5000x: (a) without superplasticizer and (b) with superplasticizer

Figure 16. Effect of superplasticizer dosage in air content of fresh grout (NHL5+w/b=0.50)

Figure 17. Isoresponse lines for plastic viscosity (Pa.s) of NHL5 grouts with for water/binder ratio of 0.50

Figure 18. Isoresponse lines for yield stress (Pa) of NHL5 grouts with for water/binder ratio of 0.50

Figure 19. Isoresponse lines for compressive strength (MPa) of NHL5 grouts at 28 days

Figure 20. Isoresponse lines for flexural strength (MPa) of NHL5 grout at 28 days