Universidade Nova de Lisboa
Faculdade de Ciências e Tecnologia
Departamento de Engenharia Civil

GROUT OPTIMIZATION FOR MASONRY CONSOLIDATION

ANA MARGARIDA ARMADA BRÁS

Tese apresentada para a obtenção do grau académico de Doutor em Engenharia Civil na especialidade de
Reabilitação do Património Edificado, pela Universidade Nova de Lisboa, Faculdade de Ciências e Tecnologia

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The people who get on in this world
are the people who get up and look for the
circumstances they want, and,
if they can't find them, make them.

George Bernard Shaw

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Resumo

A injecção com caldas ("grouts" em inglês) é uma das técnicas correntemente utilizadas na consolidação de alvenarias de pedra. O objectivo desta técnica é aumentar a compacidade da alvenaria, tirando partido do elevado nível de porosidade dos seus materiais, criando ao mesmo tempo uma ligação entre os panos interior e exterior. Deste modo, melhora-se não só a resistência ao corte e à flexão como também à compressão, permitindo que as cargas verticais e horizontais se distribuam de modo mais uniforme pelas três zonas da alvenaria.

A qualidade da consolidação depende quer dos materiais existentes, quer do desempenho geral da calda utilizada. As propriedades da calda no estado fresco revelam-se tão importantes quanto as que atinge no seu estado endurecido, na medida em que o seu desempenho global depende da capacidade da calda em penetrar na porosidade aberta. Consequentemente, a reologia das caldas deve ser compreendida e controlada, de modo a que possam ser bombeadas e fluam no meio poroso onde estão a ser injectadas. De facto, as caldas no estado fresco são um material com um comportamento que se pode considerar intermédio entre o de um fluido e o de um aglomerado húmido de partículas. Deste modo, poderão ser aplicados conceitos da reologia para a sua caracterização em função de propriedades intrínsecas reológicas como a tensão de cedência e a viscosidade que permitem avaliar e prever o comportamento das caldas.

O trabalho realizado visa o desenvolvimento de uma metodologia que permita a optimização das caldas de injecção (atendendo a que na prática há um certo empirismo na sua formulação e avaliação) com vista à sua aplicação em consolidação de alvenarias de pedra. A definição de um procedimento adequado que optimize a calda para melhorar o seu desempenho é fundamental. Como tal, foram testadas diferentes formas de mistura de caldas e ordem de colocação de componentes. Constatou-se que existem notórias diferenças no comportamento de uma calda misturada de formas diferentes e que essa forma de mistura pode ser optimizada. Foram analisadas as propriedades das caldas de injecção no estado fresco e endurecido, procurando perceber quais os parâmetros que têm impacto no seu comportamento e de que modo se pode optimizar uma composição. Para além do estudo das propriedades das caldas no seu estado fresco e endurecido, a interacção entre a calda optimizada anteriormente e a alvenaria assume importância substancial, pelo que foram também realizadas injecções em pequenos provetes numa tentativa de estabelecer critérios que contribuam para uma selecção adequada de pressões de injecção e de composições de caldas para reforço de alvenarias.

Com vista a estudar a adequabilidade do cone de Marsh (correntemente utilizado na avaliação em obra da fluidez de uma calda de injecção), simulou-se numericamente o escoamento de caldas no cone e comparou-se com valores experimentais com o objectivo não só de encontrar uma relação entre os resultados experimentais e numéricos, procurando calibrar o modelo, como também procurar relacionar o tempo de fluidez com os parâmetros reológicos.

Summary

Old masonries such as heritage or modest buildings represent a large portion of the construction area in many urban centres, such as Lisbon, Portugal. They frequently present a bad state of conservation and often require the need for consolidation in order to increase or maintain the structural characteristics. The purpose of a grout injection technique applied in a multiple leaf wall is to increase the compactness and create links between the internal and external leaves, which improve not only shear and flexural resistance but also the compressive strength. Therefore, consolidation quality depends upon the characteristics of masonry materials and on grout behaviour. Fresh grout properties seem to be as important as the ones in the hardened state since grout consistency is an essential characteristic to allow the filling of voids. Thus, the optimization of grout injection is a task of major importance.

The grout rheological behaviour is connected to its fresh properties. Therefore, rheology is a major tool for grout development since it enables not only the understanding of interactions between different ingredients, but also it may be used for quality control and grout design.

The research work aim was to develop an optimized methodology of grouts for masonry injection and consolidation. Before the development of the research, an extensive study concerning the state of the art on this subjected was made. Those notes are presented in Chapter 1 to 3 and they represent the tools needed for research development.

Grouts are highly complex materials and a complete understanding of the structural behaviour on the macro scale (injected masonry) requires a detailed understanding of the material behaviour on the nano and micro scales. Chapters 4 to 8 present the development made on this subject. It is shown that there are differences in the grout behaviour when different mixing procedures are used and that fresh grout properties may be optimized if a proper mixing procedure is chosen. After the grout mixing procedure optimization, the second step for injection purpose should focus on optimization of grout composition and injection capacity. Thus, the research work was planned to get an insight into the rheological properties that may have an impact in grout behaviour (Chapter 5 and 6). To improve the knowledge about the physical mechanisms that take place during injection and that determine the penetration of the grout in the masonry, reproducible masonry samples of cylindrical shape were tested (Chapter 7). A comparison between the numerical simulation and experimental results of flow time (usually measured on field through the Marsh cone for grout quality evaluation) was made.

The goal was to get a good agreement between the numerical results and experimental values, in order to confirm the validity of the numerical approach and allow the use of those numerical results obtained for Marsh cone to predict grout flow time.

As the conclusion and proposals for future research are described in the end of this thesis, the material properties investigated in this research work must be incorporated in a more comprehensive study for grout development concerning several specific applications. The methodology presented in this thesis will provide part of the tools necessary for that purpose.

Notations and symbols

τ	Shear stress
F	Force
A	Area of the top of element
γ	Shear strain
t	Time
$\dot{\gamma}$	Shear rate
η	Viscosity
τ_0	Yield stress
ϕ	Ratio between the solid phase volume and the mixture volume
ϕ_m	Maximum packing fraction
ϕ_c	Critical packing fraction
η_s	Viscosity of the suspending medium
w/b	Water/ Binder ratio
w/c	Water/ Cement ratio
η_0	Viscosity at the first Newtonian region
η_∞	Viscosity at the second Newtonian region
K_2	Consistency
n	*P*ower-law index
τ_{0s}	Static yield stress
τ_{0d}	Dynamic yield stress
λ	Flocculation level inside the material (structural parameter)
θ	Characteristic time of evolution of the structure
α	Material parameter
A_{thix}	Rate of flocculation
B_3	Friction coefficient between particles
n_3	Number of particles
U_0	Initial degree of dispersion
H	Coagulation rate constant
V	Flowing speed

h	Thickness of the second layer
t_c	Critical time
η_p	Plastic viscosity
c	Insignificant constant
T_{limit}	Grout threshold temperature
η_{st}	Steady state viscosity
I	Injectability
h_{inj}	Injection height
T	Splitting tensile strength
P	Maximum applied load indicated by the testing machine
l	Length
d	Diameter
FT	Flow time
P_A	Injection pressure in point A
P_B	Injection pressure in point B
ΔP	Pressure variation
CFD	Computational Fluid Dynamic
VoF	Volume of Fluid
V_W	Volume of decanted bleed water
V_1	Volume of sample at beginning of test
ASTM	American Standard specifications and Test Methods
MIX	Mixture
EN	European Standard
DEC/UNL	Department of civil engineering of Universidade Nova de Lisboa
FCT	Faculdade de Ciências e Tecnologia
LNEC	Laboratório Nacional de Engenharia Civil
CV	Coefficients of variation
C.I.	Confidence Interval
ANOVA	Analysis of variance
QC	Quality Characteristic
DOE	Design of Experiments
SEM	Scanning electron microscope
E	Elastic modulus

Contents

List of Figures

List of Tables

The following seven papers are included in the thesis and comprise part of the experimental work performed for the purpose of this thesis:

Bras A., Henriques F. (2008) Influência das propriedades de caldas no estado fresco na sua concepção para efeitos de injecção em alvenarias antigas. Proceedings of the 4th CINPAR - International conference on Structural Defects and Repair, Universidade de Aveiro, Junho 2008.

Bras A., Henriques F. (2008) Consolidation by grout injection technique- analysis of fresh grout properties. Proceedings of the 8th International Seminar on Structural Masonry, Istanbul Technical University, Istambul, 79-87 November 2008.

Bras A., Henriques F. (2009) The influence of the mixing procedures on the optimization of fresh grout properties. RILEM Materials and Structures 42: 1423-1432.

Bras A., Henriques F. (2009) Grout mix optimization for injection purpose *-1st part of the development.* RILEM-Symposium on Rheology of Cement Suspensions - 3rd RILEM International Symposium on Rheology of Cement Suspensions such as Fresh Concrete, Reykjavik, Iceland, 19th - 21st of August, 2009. Poster communication.

Bras A., Henriques F., Cidade M. T (2010) Effect of environmental temperature and fly ash addition in hydraulic lime grout behaviour. Construction and Building Materials Journal, Elsevier, doi:10.1016/j.conbuildmat.2010.02.001

Bras A., Henriques F. (2010) Investigation of grout parameters for masonry injection, using a natural hydraulic lime binder. Submitted to International Journal of Architectural Heritage: Conservation, Analysis, and Restoration, Taylor & Francis.

Bras A., Henriques F., Cidade M. T. (2010) Effect of temperature and fly ash addition in hydraulic lime grout behaviour-shear and time dependence. Submitted to RILEM Materials and Structures, Springer.

Chapter 1. Introduction

1.1 Aim and scope of the research

The research work aim was to develop an optimized methodology of grouts for masonry injection and consolidation.

In masonry, grouts (or pastes) designate a fluid mixture of binder, water and, possibly, admixture (see 2.4). The purpose of a grout injection technique applied in a multiple leaf wall is to increase the compactness and create links between the internal and external leaves, increasing not only shear and flexural resistance but also compressive strength. The correct grout selection should optimize two often competing properties, namely injectability and stability. 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 fill a particular type of void. In practice, the variability of voids within masonry requires an ability to fine tune the rheological properties of the grout in order to optimally fill a given void. So, it was developed a methodology with the main purpose of finding not only the best mixing procedures but also the optimal grout composition from a rheological and injectability points of view.

The structural improvement of masonries is not only based on the increase of the mechanical resistances. Ductility and durability should also be taken into account since an incompatible choice of grout composition may jeopardize the performance of old masonries. However the focus was early limited to grout rheological and injection studies, selecting always grout composition based on durability and ductility criteria in order to ensure compatibility with the original materials to be injected.

It is a well known fact that old masonries such as heritage or modest buildings represent a large portion of the construction area in many urban centres, such as Lisbon, Portugal. They frequently present a bad state of conservation and have particular technical needs and specific problems that require approaches different from the ones currently used in other situations, as is the case of concrete structures.

Some historic and important masonry structures are “buildings at risk” and a correct building intervention and philosophy of repair is needed. Masonry conservation frequently requires the need for consolidation in order to increase or maintain the structural characteristics and here grout injection techniques may became an important solution.

1.2 Limitations

The numbers of parameters influencing masonry consolidation is quite large and many of them are not included in this research. The parameters included are mainly the grout material properties, the injection pressure which were chosen taking into account the masonry’s porous media to be injected.

Masonry characteristics are very different; some of them present only a single leaf and others multiple leaf walls. The connection between the leaves may be different as well as the filling material of the internal leaf. In the case of a multiple leaf wall, the section is composed by two resistant external leaves and an internal layer filled by small stone pieces, sand, mortar or other kind of unbounded material. The absence of cohesion among elements, the existence of voids and cracks as well as the deficient connection between leaves lead to a masonry non-monolithic behaviour. This means that the wall becomes brittle, namely under vertical and horizontal loads. Therefore, shear and flexural strength are affected and deformation and failure of the two external leaves may occur, which indeed justify the use of grout injection technique for consolidation.

The aim of the laboratory research on grouts for injection is to define a right composition for grouts to be used for the strengthening of walls and to improve the knowledge about the physical mechanisms that take place during injection. Even if injectability tests are carried out on site, the characteristics of the grouts should be fine tuned in laboratory. Since it is hard to reproduce a real masonry and it is difficult to visualize what is happening inside the porous media being injected by grout, masonry samples were created using specific types of sand, with several grain size distributions (in order to get a representation of the variability of voids within masonry). So, to improve the knowledge about the mechanisms that take place during injection and that determine the penetration of the grout in the masonry, transparent cylinders were filled with a fraction of crushed limestone sand and injected unidirectionally from bottom to top. These types of aggregates were adopted since they have, just like masonry, a water absorbing action.

1.3 Research objectives

The research lines proposed in this work were the following:

1. Analyze the influence of the mixing procedure in grout behaviour and how it may improve some essential injection characteristics.
2. To study the influence of environmental temperature and of the addition of fly ash (a by-product of the combustion of pulverized coal in thermal power generation that was used in some grouts since it reduces the costs and the environmental impacts) in fresh grouts behaviour. An attempt was made to achieve an optimal grout composition by using the Taguchi method.
3. To study the effect of temperature and/or fly ash in grout thixotropy and flocculation rate and to know if it is possible to regulate and reduce masonry pressure during and after grout injection if thixotropic parameters are controlled.
4. To increase the knowledge about the physical mechanisms that take place during grout injection and to define a right composition for grouts to be used for the strengthening of walls, enabling also grout design regarding several issues like best injection pressure to adopt.
5. Adopt computational modelling for grout design.

1.4 The importance of correct judgements of grouts design for masonry consolidation

Consolidation quality depends mainly on masonry materials characteristics and on grout behaviour. Fresh grout properties seem to be as important as the ones in the hardened state, since grout consistency should allow the voids to be filled. Those fresh grout properties are really important especially if the injected region is inaccessible for visual inspection (except when using destructive tests).

The grout rheological behaviour is connected to its fresh properties and so it should be well understood and controlled so that the fluid may be pumped and flow correctly inside the porous medium, enabling a grout control quality. Pumping and flow depend on rheology and thanks to an increasing scientific approach it is becoming possible to predict fresh properties, design and select materials to achieve the required performance.

The purpose of performing rheological measurements is not only to understand the interactions between different ingredients in a grout, but also for quality control of products and processing conditions. Results obtained in rheological tests can be used to design process equipment (like pumps) or to be used by a customer to determine the acceptance of a product.

1.5 Thesis content

The state of the art presented in this thesis is found in Chapter 1 to 3. At the end of the latter chapter it is found the references needed for that study. Chapter 4 to 8 present the development made on this subject and at the end of each chapter it is also found the specific necessary references. Conclusions and recommendations and Future research are presented in Chapter 9 and 10, respectively.

Chapter 2. Grout injection as a consolidation technique for masonry

2.1 Purpose

In this chapter it will be given a brief and general description of masonries, some of its typologies and damage mechanisms. Concerning masonry conservation, it will be presented the grout injection technique as one of the techniques most widely used in the consolidation of this type of buildings, to increase or maintain the structural characteristics. The definition of grouts should deal with the description of the function of the grout and for that some definitions will be presented. Concerning grout injection, there are two main categories of materials that may be used: inorganic and organic binders. The following topics present some material characteristics.

2.2 General description of masonry and causes of damage

Masonry exists for many centuries and has been in constant evolution since the placing of big stones one on top of each other to the agglomeration of fine prefabricated stones and mortar that is used today. To develop and use structural consolidation techniques, the designer must start from the study and thorough understanding of the real nature and behaviour of masonry.

Masonry is a composite material, made of stones and mortar, bricks and mortar, or stones, bricks and mortar. Not only the nature of mortar and stones can account for the type of deterioration of the masonry, also the structural built up plays an important role (Van Rickstal, 2000), (Van Gemert *et al.*, 1997).

Most buildings were erected using the stones available in the neighbourhood such as granite, marl, limestone or sandstone. Those natural stones withstand poorly the effect of acid rain (especially the last ones). When the replacement of natural stones is necessary, they should be similar to the original stones, with the same composition, the same porosity providing similar properties with regard to water transport and the same frost resistance.

Bricks are artificial stones made by baking clay at high temperature and have, in course of time, reduced the use of natural stone for masonry. Brickwork is often combined with masonry of natural stone, especially in valuable monuments since natural stones gave the building an image of wealth and were known to be very durable. The replacement of natural stones can be problematic if the quarries are no longer exploited. Besides, many natural stones undergo an accelerated weathering because of air pollution. The original stones and the replacing stones have a different structure and because of small differences in the weathering resistance, the homogeneous outlook of the facade is disturbed. The fabric of natural stones in the old artisanal way is expensive. The knowledge, the tools and the workers are not readily available.

Mortar consisting primarily of lime and sand, has been used as an integral part of masonry structures for thousands of years. Traditional mortar was made from lime putty, or slaked lime, combined with local sand, generally in a ratio of 1 part lime putty to 3 parts sand by volume. Often other ingredients, such as brick dust, clay, natural cements, pigments, crushed sea shells (also a source of lime) and even animal hair were also added to mortar. However, the basic formulation for lime putty and sand mortar remained unchanged for centuries until the advent of Portland cement or its forerunner, Roman cement, a natural hydraulic cement. In the 1930s more new mortar products, intended to accelerate and simplify masons' work, were introduced. These included masonry cement, a premixed, bagged mortar which is a combination of Portland cement and ground limestone, hydrated lime, machine-slaked lime that eliminated the necessity of slaking quicklime into putty at the site (Van Rickstal, 2000).

$$CaCO_3 + heat(\pm 900^{o}C) \Rightarrow CaO + CO_2 \tag{2.1}$$

$$CaO + H_2O \Rightarrow Ca(OH)_2 \tag{2.2}$$

$$Ca(OH)_2 + CO_2 \Rightarrow CaCO_3 + H_2O \tag{2.3}$$

During the production of hydrated lime several chemical reactions take place. The calcium carbonate (that could be found in lime stone or in shells) is dissociated at high temperature (Eqn. 2.1). Then, the calcium oxide is slaked with water originating calcium hydroxide (Eqn. 2.2). Reaction (Eqn. 2.3) is using CO_2 and hence requires the presence of air. For a thick wall, this is always a problem.

To achieve some hydraulic properties to lime mortars, natural or artificial pozzolans should be used. A pozzolan is an amorphous and finely-divided material that reacts with calcium hydroxide, in the presence of water, to form compounds possessing cementitious properties. They are present on earth's surface such as diatomaceous earth, volcanic ash, among others, or are produced when pulverized coal is burned in electric power plants (like fly ash). The hydraulic properties gave these mortars a good early strength development.

However, the use of the relatively soft air hardening lime mortar gave ancient masonry a good capacity to recover from settlements. The lime mortar has a slow strength development and for historical buildings that were constructed slowly, this was not a big disadvantage. On the contrary, deformations during the construction were distributed and moderated the stresses. Besides that, the mortar remained less strong than the stones and occurring cracks were located in the mortar joints, where they could easily be hidden by repointing. Generally, a lime mortar is more elastic and this provides an additional safety with regard to differential settlements. Lime mortar contains no or hardly any sulphate or alkalis. This reduces the risk for salt efflorescence.

The previous statement implies that there are two main reasons to use mortars that are compatible with the original mortars. The first one is the fact that using a modern mortar would result in introducing a component that is harder than the old mortar and in most cases also harder than the stones that were used. Settlements become hard to follow and bending/splitting action caused by hard cement mortar among soft lime mortar may occur. According to van Rickstal, the new hard cement mortar among soft lime mortar splits the masonry just above the hard zone since the zone on the left and right hand side of the new mortar are softer and more deformable (Fig. 2.1). Besides that, a cement mortar has a different porosity causing a different action with regard to water transport.

The second main reason to use mortar compatible with the original ones regards what is mentioned in the Venice Charter (Venice Charter, 1964) is to use mortars that imitate the original mortar and that are as hard as the original one or even somewhat softer. Obviously these statements will regulate grout composition to apply in masonry.

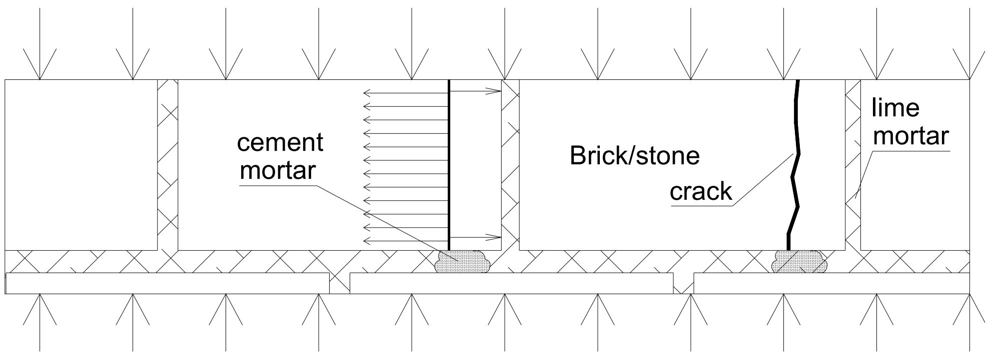

Figure 2.1: Crack formation due to bending/splitting caused by hard cement mortar among soft lime mortar.

Also the repointing action should be carefully adopted in masonry joints since it is often an important action in the restoration of facades. An appropriate mortar should be adopted. The aim of this repair is to bond the stones of the external leaves, particularly in the case of badly bonded irregular stones and to obtain an external confinement of the wall in order to increase the masonry shear strength and avoid water penetration. The repointing can be carried out also in conjunction with grout injection.

When disregarding the original composition, the new mortar used for repointing is almost impermeable to water vapour in comparison with the original situation. Therefore, the water vapour concentrates behind the repointed layer and salt crystallization or frost can then easily push the new layer outwards causing even more damage since the adhesion of the repointed mortar to the existing bricks or stone is very good. Generally, repointing should be limited to the damaged parts, using a mortar composition that corresponds as well as possible with the mortar used for the original pointing. Very often, a consolidation injection is combined with a partial or general repointing (Van Rickstal, 2000**)**, (Courradi *et al.*, 2008).

There are several causes of damage to masonry. Some of them were already presented. Generally they could be divided into: physical and physico-chemical mechanisms; biological mechanisms and mechanical damage. Quite often physical and physico-chemical mechanisms are induced by the presence of water, both in liquid or vapour forms and mostly need a long period of time to cause visible damage. The presence of moisture depends of the location and environmental conditions of the masonry, while transport is a function of porosity and pore sizes of the masonry.

Moisture transport inside the masonry is a potential cause for lixiviation, a phenomenon that may cause a decrease of internal cohesion. Freeze-thaw action or the presence of hygroscopic salts may cause physical damages due to the increase of volume induced by crystallization. When water is inside masonry, hydrofobic treatment should not be applied. The outer layer of the masonry becomes impermeable to water. Only water vapour is able to be transported. This means a much slower process of loss of water then when the water is able to proceed to the surface itself. In this way two phenomena take place: spalling off of the treated outer layer of masonry caused by hygroscopic salts growth due to the slower transport of water inside; decrease of the internal cohesion since moisture movements give rise to the dissolution and the corrosion of the binder (Van Rickstal, 2000**)**, **(**Barbara Lubelli *et al.*, 2004).

Grout injection is very suitable to repair this kind of damage. The more uniform the grout fills the voids caused by the erosion of the binder, the better the final consolidation.

Atmospheric pollution has also a great impact on masonry given the fact that new ions may be added to those already existing inside the masonries, thus increasing the risk of physical or chemical damages. Corrosion of metallic elements may also occur, inducing fissures and cracks in masonries or colour changes.

Concerning the biological damage, the formation of algae, fine roots of plants or the micro organisms that bring bacteria that produce nitrates and sulphates as residue of their metabolism, are also some of the possible damage mechanisms.

Mechanical damage may be caused by mistakes in the original design of masonry or even by a different use of the building, for instance a dwelling building converted to a gymnasium (were the load is much higher than the designed load). However, those problems are not only due to human actions. Earthquakes, wind, rain or storms can cause severe damage. Mechanical damage is very suitable to be repaired by grout injection.

Masonry types are very different; some of them present only a single leaf and others multiple leaf walls and these cannot be compared to brick or regular stone-masonries. The connection between the leaves may be different as well as the filling material of the internal leaf. In the case of a multiple leaf wall, the section is composed by two external leaves and an internal layer filled by small stone pieces, sand, mortar or other kind of unbounded material (Fig.2.2).

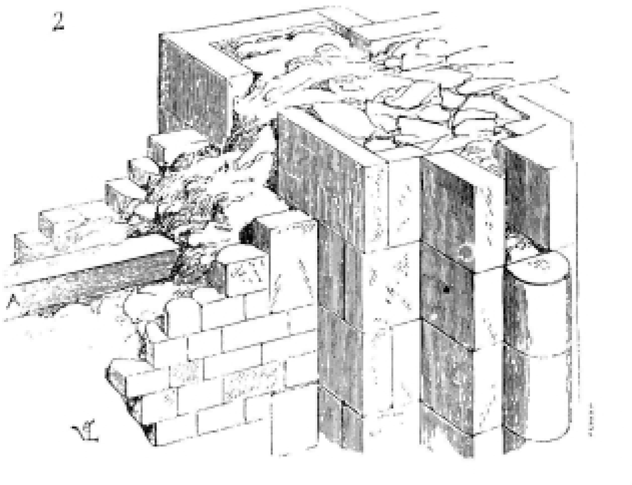

Figure 2.2: Example of a section of a three leaf masonry wall according to Viollet-le-Duc.

The absence of cohesion among elements, the existence of voids and cracks as well as the deficient connection between leaves lead to a masonry non-monolithic behaviour. This means that the wall becomes brittlier, namely under vertical and horizontal loads. Therefore, shear and flexural strength are affected and deformation and failure of the two external leaves may occur. Those leaves tend to separate (Fig. 2.3) and a partial or total collapse of the wall can take place (out-of-plane mechanisms). This mechanism of collapse has frequently occurred during seismic events.

One of the most difficult tasks for engineers and architects is the repair and/or retrofit of this damage. Traditional masonry works are also particularly susceptible to in-plane shear actions due to their very low tensile strength, so reinforcement should prevent both out-of-plane and in-plane collapse mechanisms (Fig. 2.4 and 2.5) (Courradi *et al.*, 2008**)**, (Penazzi *et al.*, 2001).

Figure 2.3: Damage detected in a masonry building repaired by introduction of concrete tie beams. Out-of-plane mechanism: separation of leaves. (Courradi *et al.*, 2008).

Figure 2.4: In-plane shear failure of the masonry walls (Decanini *et al.*, 2004).

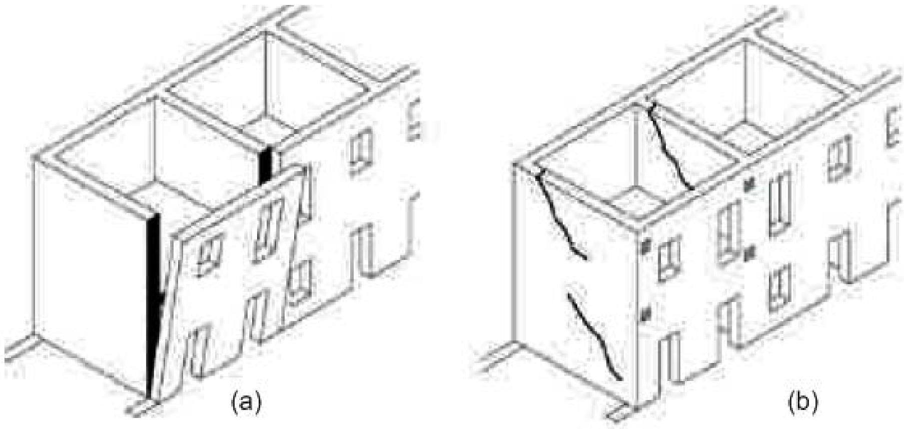

Figure 2.5: Damage modes in masonry buildings. Out-of-plane masonry wall failure (a) and in-plane (shear) cracking (b). (b) shows wall ties securing the front facade from out-of-plane failure, which then imparts a greater load to the in-plane walls, initiating cracking (Decanini *et al.*, 2004).

Masonry conservation frequently requires the need for consolidation in order to increase or maintain the structural characteristics. Grout injection is one of the techniques most widely used in the consolidation of this type of buildings, to improve the masonry shear and flexural strength. The purpose of this technique is to increase the compactness of the masonry and, at the same time, create links between the internal and external leaves, therefore increasing its resistance and the monolithic behaviour.

According to Schueremans *et al.* (Schueremans *et al.*, 1998), the grout injection technique increase masonry reliability, since one of the main goals is to improve the average strength and/or reduce the variance on the strength between different masonry sections. However, the grouts to be used for the injection should be correctly designed to achieve the maximum performance. It means that besides strength, principles such as masonry ductility and durability should be taken into consideration since a bad choice of grout composition may jeopardize the performance of old masonries.

The efficacy of consolidation techniques depends upon masonry materials characteristics and on grout behaviour in fresh and hardened state. The optimization of grout injectability involves an adequate control of fresh properties, namely: the rheological properties (so that the fluid may be pumped and flow inside the porous medium), water retention and bleeding, among others, which allows the grout to flow easily inside the porous media avoiding that an excessive loss of its mixing water may occur as a consequence of the medium absorption characteristics.

In this way, the grout specification involves a previous verification of the following items:

1. Grout flow capacity inside the porous media;
2. Grout composition compatible to the original materials used in masonries.

This flow capacity and the ability to fill the voids, together with a suitable grout composition, will regulate the consolidation quality.

2.3 Injection technique

Grout is a binder employed for the filling, homogenization, imperviousness, consolidation and/or upgrading of the mechanical properties of systems presenting pores, voids, cracks, loss of cohesion or of cohesionless systems (like masonry) (Toumbakari, 2002). The binding agent will cure and increase the internal cohesion of masonry, increasing also its monolithic behaviour, together with the load capacity.

Since the grout is introduced into the internal part of masonry it does not damage the aesthetical outlook of the building. However, grout injection is not reversible but when materials are used that comply with the original materials, it is an acceptable technique in accordance with the Venice Charter (Van Rickstal, 2000).

The use of fluid grout avoids dismantling and rebuilding defective masonry in many cases and there is a choice of three basic methods for grout injection technique: gravity systems; hand or mechanical pumped systems and vacuum systems. The nature and condition of masonry selects the best method. Gravity technique is particularly suitable where the masonry is very vulnerable to movement under pressure. Pumped systems of various kinds may be used to deal with most grouting problems. Vacuum systems may be useful where fine fractures and small-scale voids are suspected (Ashurst, 1990).

An injection installation generally consists of a complete set of device such as: mixing installation (that mix the materials to be injected); collector (which collect and stirs the grout previously mixed); pumping installation (which is fed by the collector and provide pressure control); conduits (flexible tubes that lead the grout from the ground installation to the injection hole). For longer distance between pumping installation and injection holes, a return conduit should be present. If there is no return conduit, the grout will stand still in the main conduit while switching from one injection hole to another. This will cause the time depending features of the grout to occur: instability and thixotropy. It depends on the quality of the grout to what extent these phenomena will arise.

Before conducting an injection the diagnosis of the masonry should be made and for that, non destructive and slightly destructive techniques are applied. Non destructive tests are very suitable to obtain a qualitative picture of the masonry structure and to decide about consolidation and for the quality control after injection. They are ideal to compare the initial state with the injected one. Among the slightly destructive techniques, coring is probably the most frequently used. The coring enables furthermore to judge the quality of the inner masonry by visual inspection of the cores. Compressive and splitting tests can be performed on the cores to get an idea of the mechanical properties of the masonry. Eventually the core hole can be inspected using an endoscope. There are several techniques but it is not the purpose of this thesis to develop this subject.

Nowadays there are also numerical and probabilistic methods that have been in development to quantify the need, the benefit and the efficiency of an injection.

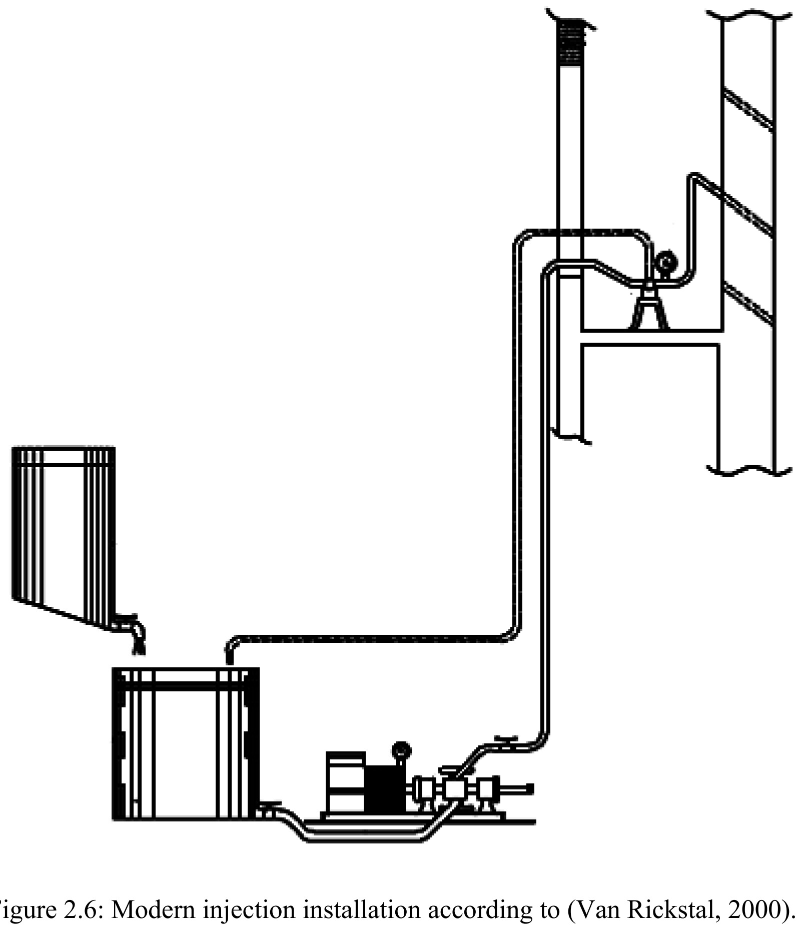

Figure 2.6: Modern injection installation according to (Van Rickstal, 2000).

The preparation of masonry obeys to several issues. Once the areas to inject are determined, the building is prepared for injection and for that masonry should be sealed to prevent the leakage of the grout. Therefore, a general repointing is recommended. Besides, a general deep repointing can be seen as a very effective structural intervention. The repointing as such already consolidates the masonry and must be fairly porous to not absorb the water of the injected grout (Courradi *et al.*, 2008), (Ashurst, 1990).

Nevertheless leakage can still occur and should then be stopped using quick-setting cement cleaned immediately. Leakages prevent to build up pressure inside the flow channels. If the stability of the masonry is very doubtful and if hydrostatical pressure is feared to arise, an external reinforcement can be justified.

After repointing, the second part of the preparation is the drilling of the injection holes. Generally they are drilled in the joints and in this way they will be less visible afterwards. The holes should incline downwards and placed according to a certain pattern, density and depth. Those parameters depend on the type of masonry, the overall condition of the masonry, the rheological properties of the grout and incidence of cracks. A zone with many cracks will be easier to inject and hence the pattern in this zone can be less dense. On the other hand the applied pressure in this zone should be lower. Therefore and for reasons of simplicity the density is often kept constant for the whole structure. Existing major cracks can easily be used for injection.

Two possible geometries for injection are presented in Figure 2.7 and 2.8. Each circle represents the injection area of a hole. The closest pattern increases the covered surface by injection compared to the square pattern.

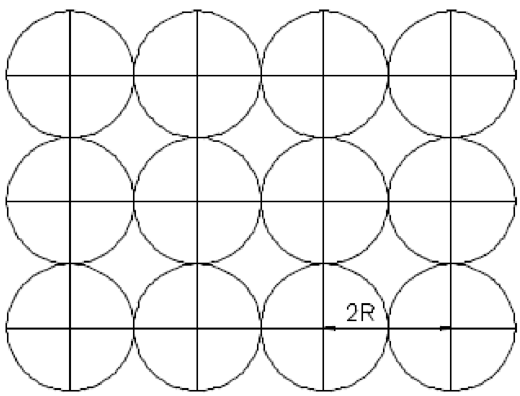

Figure 2.7: Injection geometry using square pattern (21.5% not covered) (Van Rickstal, 2000).

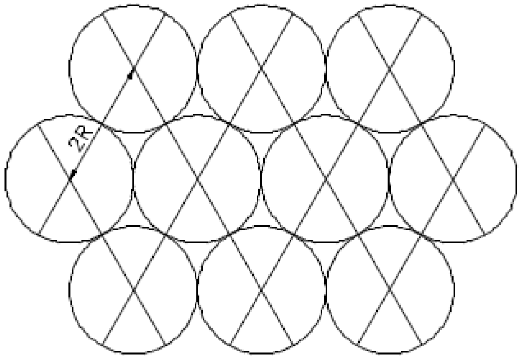

Figure 2.8: Injection geometry using closest pattern (9.3% not covered) (Van Rickstal, 2000).

The recommended number of holes per square meter found in literature is mentioned in Table 2.1. The density of the injection holes is expressed as holes per square metre.

Table 2.1: Injection holes pattern with some recommendations found in recent literature.

Reference	nº of holes/m^2	Comments
(Binda *et al.*, 1991)	2	The holes should at least reach the 2/3 of the wall
(Baronio *et al.*, 1992)	-	2-4 holes/m^2 is not enough. The covered area for one injection hole depends on the penetration of the grout inside the masonry
(Van Rickstal, 2000)	-	Closest pattern of the injection holes is preferable, density can vary depending on the quality of the masonry
(Toumbakari, 2002)	4,5	Slightly inclined holes, preferentially drilled inside the mortar and not in the stones or bricks
(Valluzzi, 2005)	11, 12	Only 70% of these holes were injected. The remaining 30% was used to check the diffusion of the grout during the intervention

During injection, when a grout reaches a large void, no pressure can be built up in the neighbourhood of that void. Due to this low pressure, the grout will enter the fine cracks only over a short distance. Thixotropy, water absorption and instability of the grout cause the blocking for further injection in these finer cracks. When the large void is finally filled, the pressure can increase again, but too much water of the grout is absorbed in the fine cracks to restart flowing. The zone hidden by the finer cracks will never be injected through this one hole and because of that the distance between holes should be carefully chosen, taking into account not only porous media properties but also grout properties and injection pressure (Van Rickstal, 2000), (Toumbakari, 2002), (Bras *et al.*, 2008).

The injection pressure is the pressure at the inlet towards the injection holes and that enables the penetration of the grout inside the masonry. The higher the pressure the easier and faster the grout will pass and because of that, the grout will loose less water by absorption and the particles will remain better in suspension. However, the pressure is limited to a few bars since the internal pressure of the grout introduce tensile stresses that can not be taken by the masonry. Increasing the pressure would soon cause additional damage to the structure. The internal pressure in masonry is the addition of the hydrostatic pressure (proportional to the height of the injected grout column that is still in fluid state) and the injection pressure (controlled by pump).

Therefore, it is recommended not to inject the holes in a vertical order. Typical compressive stresses in ancient masonry are about 1 MPa and tensile stresses are close to zero so, internal pressure might cause big tensile stresses or might push out the outer leaf of the masonry. Generally, the injection stop criteria for one hole in masonry are the following: the pressure exceeds permanently the injection pressure; the grout emerges freely at adjacent injection holes; a predetermined quantity of grout is injected in that hole. In Table 2.2 some recommended values for the injection pressure are given.

Table 2.2: Injection pressure values for masonry found in literature.

Reference	pressure (bar)	Type of grout
(Courradi *et al.*, 2008)	1.0	Hydraulic lime based grout (w/b=?)
(Courradi *et al.*, 2008)	1.0	Albaria Iniezione 100
(Binda *et al.*, 1997)	0.2 to 0.6	Micro fine cement based grout (w/b=1.5)
(Binda *et al.*, 1997)	0.2 to 0.6	Hydraulic lime grout (w/b=0.80)
(Binda *et al.*, 1997)	0.2 to 0.6	Hydrated lime mixed with dust bricks (w/b=?)
(Keersmaekers *et al.*, 2006)	1.0	CEMIIIA 42.5+$Ca(OH)_2$ (w/b=0.675)
(Valluzzi, 2005)	0.5	Hydraulic lime based grout (w/b=0.55)

Binda and colleagues in (Binda *et al.*, 1997) suggest a type of grout able to be injected in a certain porous medium. The following table (Table 2.3) is based on those results. It can be observed that the presence of silt and clay make the injection more difficult, due to their very low fineness. Experimental results show clearly that while gravel and sand are injectable, silt and clay can only be injected up to a certain point with acrylic resins. However, they have inconveniencies, like poor bond to wet surfaces, low resistance to thermal stresses. Besides that, exothermic phenomena can take place when resins are injected in large voids, causing damages to the wall itself.

Table 2.3: Injection capacity of different grouts in a certain porous media.

Grouts		gravel	sand			silt	clay
			coarse	medium	fine		
Suspension	microfine cement						
	cement						
	bentonite						
	sodium silicate cement						
Solution	acrilic resins						
	grain size (mm)	2	0.5	0.25	0.074		0.005
	injection capacity	→					higher

2.4 Grout design for masonry: an overview

There are several definitions of grout in cement and concrete literature. In the context of masonry (Ashurst, 1990) defines grouting as the introduction of a binding agent in the form of liquid into masonry or soil. (Toumbakari, 2002) defines grout as a binder employed for the filling, homogenization, imperviousness, consolidation and/or upgrading of the mechanical properties of systems presenting pores, voids, cracks, loss of cohesion or of cohesionless systems. According to (Vintzileou, 2006) grout may be defined as a binder (either inorganic or organic) injected to masonry with the purpose to fill cracks, voids and (to some extent) pores of the in situ materials. In conclusion, the definition of grout should deal with the description of the function of the grout.

Over the past two decades, grouts (especially cement based) have been used for an increasing number of structural purposes, with more and more demands being placed on the grout. Many of these developments have been made possible by the use of cement replacement materials such as: silica fume, pulverised fly ash, blast furnace slag, metakaolin, admixtures, among others (Sonebi, 2006), (Fernàndez-Altable *et al.*, 2006), (Mirza *et al.*, 2002), (Van Gemert *et al.*, 1997) which are now widely accepted as giving economic and technical advantages, taking also into consideration durability issues.

Concerning grout injection, there are two main categories of materials that may be used: inorganic and organic binders. The following topic presents some materials characteristics.

2.4.1 Inorganic binders

Binders obtained from inorganic raw materials include hydraulic binders such as: hydraulic lime(s), ordinary Portland cement and all other types of modern cements, lime-pozzolan mixtures; and air-hardening binders, such as hydrated lime.

Hydrated lime grouts cannot contribute to the repair and even less to the strengthening of masonry since they require the presence of carbon dioxide for hardening. It is known that the diffusion of air at the interior of the masonry mass is a slow process. This leads to a very slow hardening and, consequently, a very slow increase of the mechanical properties of this binder (sometimes hardening does not even occur).
On the contrary, hydraulic grouts present a very interesting option for the composition of injection grouts since they are both from the chemical-physical and mechanical point of view more adequate for use in historic masonries, provided that they are adequately designed.

Hydraulic binders may be subdivided into two main categories, namely cement and hydraulic lime based grouts. Cement based grouts were initially pure cement grouts. However, it was proven that their injectability properties were inadequate for filling the small size voids and cracks of historic masonries because of clogging (Eklund *et al.*, 2008), (Axelsson *et al.*, 2009), (Vintzileou, 2008)). Due to this behaviour Miltiadou in (Miltiadou, 1990) studied the addition of ultra fine materials to cement material (on the basis of specific granularity criteria).

In this way, grouts of high injectability and adequate mechanical properties were reached at the same time. In order to serve the specific needs of each historic structure there is the need for a wide range of mechanical properties of grouts to be available. Thus, binary grouts (mixes of cement and hydrated lime, natural or artificial pozzolans, silica fume, etc.) and ternary grouts (cement, hydrated lime and natural or artificial pozzolans) were developed.

Grouts with cement percentage varying mainly from 50% to 75% reach compressive strengths varying between 10 to 30 MPa with a tensile strength range of 1.2 to 3.0 MPa (Miltiadou, 1990) and they proved to be efficient in enhancing the mechanical properties of masonry to which they are injected (Vintzileou *et al.*, 1995). According to the previous paper, the vulnerability of three-leaf masonry to compression loads is confirmed and early separation of external leaves from the internal filling material may rapidly lead to the disintegration of the wall. So, the injection of cement based grouts increase the compressive strength of wallets by 50% to 200% in respect to the initial one.

However, cement grouting led to stiffness increase values, which may increase structural damage. It is known (Vintzileou, 2006) that mechanical tests did not confirm the need for grouts with high cement content. Furthermore, the use of grouts with reduced cement content would be beneficial for the protection of mosaics, frescoes and decorative elements on masonry surfaces, as physico-chemical incompatibility with the in situ materials is prevented. Thus, enhanced durability of the intervention is expected. All this led to the development and investigation of alternative mixes, namely ternary grouts with reduced cement content.

Ternary grouts composed of more than two constituents, such as cement in a reduced percentage (generally 10% to 50%), lime, pozzolans (natural or artificial), ultra fine materials (such as fly ash or silica fume), were developed (Toumbakari, 2002), (Vintzileou, 2008).

In Miltiadou-Fezans *et al.* work (Miltiadou *et al.*, 2006) the design of high injectability grouts was performed following performance requirements based on masonry structural needs. Therefore, the following target values were set for the basic mechanical properties of the grouted masonry: tensile strength approximately double that of masonry before grouting and compressive strength approximately equal to 3.0 MPa. Besides that, the physical–chemical properties of the raw materials should be selected such that the durability of the structure and its precious mosaics would not be jeopardized. Finally, the grouts should be injectable enough to fill fine voids and cracks (estimated minimum nominal width of voids and cracks equal to 200 μm).

Based on the aforementioned requirements, six grout mixes were designed and tested (to assess their physical, chemical and mechanical properties). The two best options where chosen for use in masonry mechanical test: a ternary grout (white cement, lime, pozzolan) and a natural hydraulic lime (NHL) based grout. The mix proportions of the selected grouts, along with their mechanical properties and injectability characteristics (penetrability, fluidity, stability) are summarized in Table 2.4 and 2.5.

Table 2.4: Mix proportions of selected grouts (%wt) and injectability characteristics (Vintzileou, 2008).

Ternary grout	White Danish cement	Lime	Pozzolan (dmax < 75 μm)	Superplasticizer SP1	Water	T_{36} (s) Sand column 1.25/2.50 mm (voids 0.2–0.4 mm) (NF P18-891)	Flow time (s) (Marsh cone test d=4.7mm)	Bleeding
	30	25	45	1	80	19	20.5	2%
NHL5 grout	NHL5			SP2	Water			
	100			1	80	22.5	22	3%

Table 2.5: Grouts mechanical properties (Vintzileou, 2008).

	Compressive (f_{gc}) and flexural (f_{gt}) strength (MPa)					
Ternary grout	f_{gc} 28d	f_{gt} 28d	f_{gc} 90d	f_{gt} 90d	f_{gc} 180d	f_{gt} 180d
	4.08	2.11	8.16	2.29	10.6	3.13
NHL5 grout	2.82	2.47	4.5	2.52	6.36	3.87

The standardized sand column test method was applied to check the penetrability and the fluidity of grouts. However, before the application of the selected grouts to the specimens, their injectability was checked and confirmed by its application to cylinders made up of filling material. Both mixes proved to satisfy the requirements of sufficient mechanical properties and injectability to fine cracks and voids.

Toumbakari work (Toumbakari, 2002) shows that it is possible to limit Portland cement content by applying lime-pozzolan-cement grouts instead of cement based ones. She concluded that it is possible to develop lime-pozzolan-cement grouts with the same injectability properties as the cement ones but also with less alkali sulphate and aluminium ions and progressive densification of the matrix due to the pozzolanic reaction. The presence of pozzolans is not only important for durability but also, by binding the portlandite crystals to the matrix, it enhances the mechanical properties of the system.

Research concerning the use of hydraulic lime based grouts (pure hydraulic lime or in combination with a pozzolanic material) is quite recent (Vintzileou, 2006). Their similarity with the in situ materials may offer a promising solution as long as they also prove to be mechanically efficient.

(Valluzzi, 2005), (Vintzileou, 2008) developed grouts with a compressive strength between 2.5 and 6.0 MPa (at the age of 1 to 6 months). (Miltiadou *et al.*, 2006) developed hydraulic lime grouts for application to a byzantine monument. At the age of six months, a compressive strength of 6.4 MPa was reached, whereas the flexural strength of this grout was equal to 3.9 MPa. Those grouts proved to satisfy the requirements of sufficient mechanical properties and injectability to fine cracks and voids.

For this reason, hydraulic lime based grouts with or without pozzolans were studied in this research. The replacement of a certain amount of the hydraulic binder by fly ash will improve grout durability. In the presence of water, pozzolanic materials react with calcium hydroxide, a basic component of hydrated natural limes, to form silicates and aluminates. Probably the calcium hydroxide consumption by fly ash minimizes the risk of ettringite and thaumasite formation. According to Collepardi (Collepardi, 1999), when hardened grouts are exposed to sulphate attack in the presence of moisture (which may occur in masonry walls of historic buildings) the resulting products may cause material expansion and loss of cohesion, that damage the grout and weakens its structure.

Detailed information concerning material characteristics will be presented in Chapters 4-8.

2.4.2 Organic binders

In this category, the main materials used for injection are polymer systems. They are the most recent binding agents used for consolidation injections. Polymers are pure liquids (no dispersions like all the other binding agents used for injection purposes) and show a relatively low viscosity and low yield stress. Since a polymer is a pure liquid, it will not suffer from thickening due to water loss. The wide range of viscosity values and the absence of particles that might hinder the flow, make polymers very suitable for injection. Moreover, polymers show a very good adhesion to dry surfaces and have a high compressive strength and, even more important, a high tensile and adhesion strength. This makes them very suitable for particular cases were the penetration of hydraulic grouts is problematic.

The polymer materials most used in restoration works can be categorised as follows: epoxy resins, polyurethane resins, methacrylic resins and unsaturated polyester resins. Organic binders may be of high injectability, they may exhibit excellent bonding properties with in situ materials and they were proven efficient from the mechanical point of view in some cases. However, their use is not recommended in case of historic masonries since they present exothermic reaction during setting that induces mechanical actions to the surrounding old materials; their behaviour under temperature variations, as well as their mechanical properties are substantially different than those of masonry and their bonding properties pronounced deterioration occurs when they are applied to a wet medium (Van Rickstal, 2000), (Van Gemert *et al.*, 1997), (Toumbakari, 2002), (Vintzileou, 2006). Concerning durability effects, their long term behaviour is unknown since these products were created recently and there was still not enough time to evaluate how they evolve in time.

Thus, once again hydraulic binders meet the best characteristics for masonry application, since they are both from the chemical-physical and mechanical point of view more adequate for use, provided that they are adequately designed.

3. Rheology of hydraulic binder suspension

3.1 Purpose

In this brief introduction to rheology it will be given an explanation on some general terms and basic facts about rheological behaviour of particle suspensions such as hydraulic lime or cement based grouts.

Only little research was made concerning rheology of hydraulic lime grouts. However, since its components and hydraulic reactions are quite similar to cement, a state of art concerning manly rheology of cement grouts will be presented.

3.2 Introduction

Rheology is by definition the science of flow and deformation of matter and investigates the relations between force, deformation and time, so this can be everything from an elastic solid to a liquid (Barnes *et al.*, 2001). The study of the relations between stress and deformation rate is called viscometry. Viscosity is defined as the resistance to flow. If the resistance of a fluid against deformation is high it means that viscosity will have a high value.

Deformation of an element of fluid is shown in Figure 3.1. If a force F is applied to the top of the element (with an area $A = x \times z$) it will result in an applied shear stress τ :

$$\tau = \frac{F}{A} \tag{3.1}$$

This applied shear stress will lead to deformation, dx of the top area A during a time period dt. The relative deformation γ is called shear strain and when it is derived with the time dt, the shear rate is obtained.

$$\gamma = \frac{dx}{y} \tag{3.2}$$

$$\dot{\gamma} = \frac{d\gamma}{dt} \tag{3.3}$$

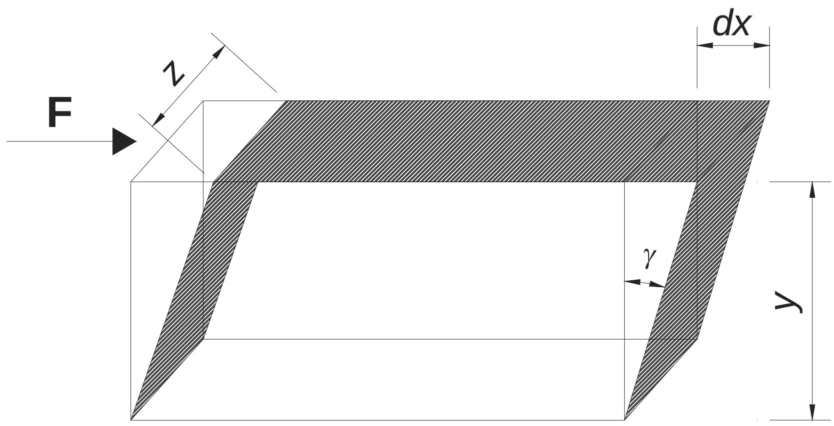

Figure 3.1: Deformation of an element of fluid due to an applied force F.

The derivate of shear stress with the shear rate gives the viscosity function $\eta(\dot{\gamma})$:

$$\eta(\dot{\gamma}) = \frac{\tau}{\dot{\gamma}} \tag{3.4}$$

If there is no dependence of viscosity on shear rate then

$$\eta(\dot{\gamma}) = \eta \tag{3.5}$$

and

$$\tau = \eta.\dot{\gamma} \tag{3.6}$$

which describes the Newtonian behaviour of a flow. As it may be observed it is analogous to the Hookean behaviour of an elastic solid since in an elastic solid material, shear strain is proportional to applied shear stress and if stress is removed, the solid material fully recovers the initial shape.

Glycerine and water are liquids which present the Newtonian behaviour. For glycerine the viscosity is equal to 1Pa.s and for water is 1mPa.s.

The following table (Table 3.1) presents the order of magnitude for viscosity values of several materials at the environment temperature of 20ºC (Barnes *et al.*, 2001), (Wallevik, 2009).

Table 3.1: Viscosity of some familiar materials at room temperature.

	Liquid	**Approximate viscosity (Pa.s)**
	Glass	10^{40}
	Bitumem	10^{8}
	Molten polymers	10^{3}
	Glycerol	10^{0}
	Olive oil	10^{-1}
	Bicycle	10^{-2}
	Water	10^{-3}
	Air	10^{-5}
w/b=0.55	Cement paste	0.4
	Cement mortar- cement:sand=1:2	4
	Concrete - cement:sand=1:2, stone:sand= 1:5	40

However not all fluids present the Newtonian behaviour. For paints, polymers, cement grouts, hydraulic lime grouts and concrete the relationship between shear stress and shear rate is non-linear (it can be shear-thinning or shear-thickening). Besides that, for some of them flow only initiates after a certain level if stress is surpassed (called the yield stress) and for others, time has large influence in the rheological behaviour due to a flocculated microstructure, where the flow characteristics are influenced by the shear history of the material (it can be thixotropic or anti-thixotropic) (Barnes *et al.*, 2001), (Billberg, 2006). This will be discussed in 3.4.

3.3 The importance of rheological measurements

The purpose of rheology was clearly summarized by O. Wallevik in (Wallevik, 2009):

1. To understand the interactions between different ingredients in a product and get an insight in the structure of the sample since there is a relation between the size and shape of particle or molecules dissolved or suspended in a solvent and the viscosity of the same solution.
2. Rheological tests are used for quality control of raw materials, processing conditions and final products. Rheological technique measures the same properties of the sample as whole and the interactions between the different components.
3. Results obtained in rheological tests can be used to design process equipment. The function of pumps and flow in pipelines is governed by the rheological properties of the substance.
4. Rheological results can be used by a customer to determine the acceptance of a product.

From a grout research point of view, the purpose of performing rheological measurements is not only to understand the interactions between different ingredients in a grout, but also for quality control of products and processing conditions. The grout should be able to be pumped and to flow inside the porous medium, which means that there is a need for quality control. Thanks to an increasing rheology scientific approach it is becoming possible to predict fresh properties, design and select materials to achieve the required performance.

3.4 Classification of flow types

Generally fluids are divided into four major groups depending on their flow properties:

3.4.1 Newtonian fluids;

3.4.2 Non-Newtonian fluids – time independent

3.4.3 Non-Newtonian fluids-time dependent

3.4.1 Newtonian fluids

As it was mentioned before, the fluids which present a constant viscosity for all shear rates are called Newtonian. Those fluids are often simple, single phase liquids and solutions of liquids with low molecular weight such as water, glycerol or oil. Silicon oils are often used as calibration fluids due to their reliability and correctness as a Newtonian fluid. Flow behaviour of Newtonian fluids is represented in Figures 3.2 and 3.3.

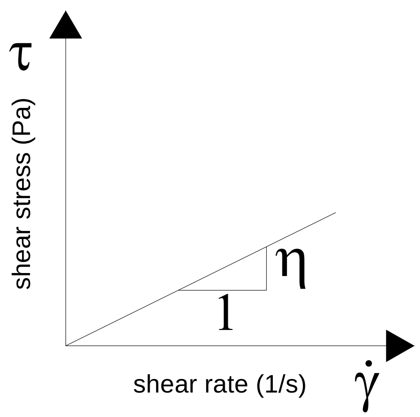

Figure 3.2: Flow curve for a Newtonian behaviour.

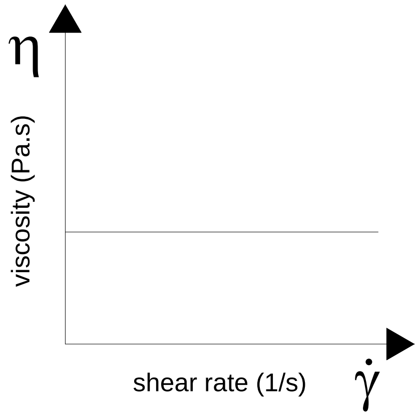

Figure 3.3: Viscosity curve for a Newtonian behaviour.

3.4.2 Non-Newtonian fluids – time independent

The Non-Newtonian fluids are those for which the viscosity depends on the shear rate applied to the sample and not on the time of shearing. This means that the sample does not have a constant viscosity- it changes with the shear rate and each value is specific for a given rate of shear. This viscosity is called apparent viscosity (Wallevik, 2009), (Hackley *et al.*, 2001).

Depending on the type of shear rate dependence these fluids may be further subdivided into three types:

- Shear-thinning (or pseudoplastic);
- Shear thickening (or dilatant);
- Viscoplastic.

The power-law expresses the simplest form of this behaviour.

Shear-thinning fluids

Shear-thinning fluids are characterised by a viscosity decrease with an increase in shear rate. This means that the power law model will then have an exponent, *n*, less than 1. Flow curve behaviour of shear-thinning fluids is represented in Figures 3.4 and 3.5.

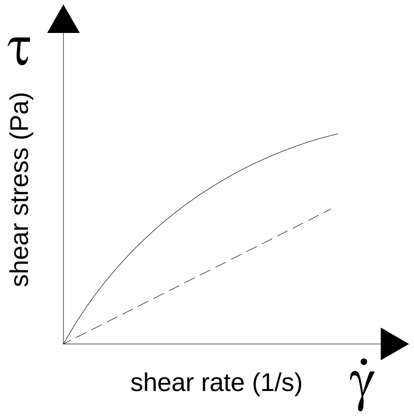

Figure 3.4: Flow curve for a shear-thinning behaviour.

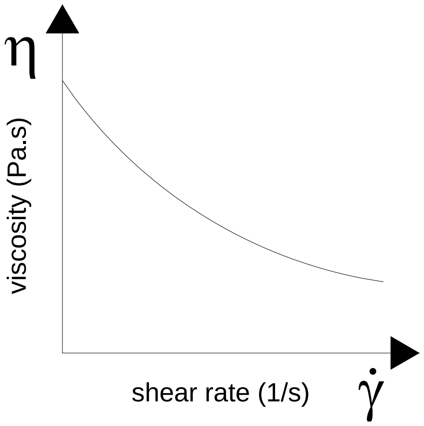

Figure 3.5: Viscosity curve for a shear-thinning behaviour.

According to (Wallevik, 2009)**,** (Barnes *et al.*, 2001), (Bras *et al.*, 2009) the main causes for shear-thinning behaviour are the result of:

<u>Orientation</u> of non-spherical particles in the direction of flow as is the case of hydraulic lime grout particles or fiber reinforced cementitious materials;

<u>Deformation</u> of spherical droplets to elliptical droplets in an emulsion, as it may happen in cement grout with air bubbles (Feys *et al.*, 2009);

<u>Disaggregation</u> of particles agglomerates. This phenomenon can occur in paints and when particles are stirred they break and paint is more easily stirred;

<u>Stretching</u> of polymer chains in the flow direction and breakage of polymer chains during flow, as is the case of polymer melt in extrusion.

Shear thickening fluids

Shear thickening fluids are characterised by a viscosity increase with an increase in shear rate. This means that the power law model will then have an exponent, *n*, higher than 1 (Barnes *et* *al.*, 2001). Flow curve behaviour of shear-thinning fluids (also called dilatancy) is represented in Figures 3.6 and 3.7.

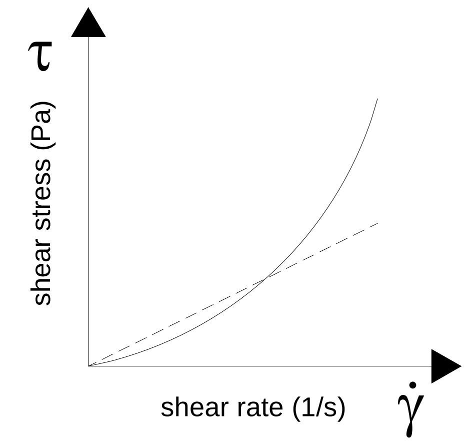

Figure 3.6: Flow curve for a shear- thickening behaviour.

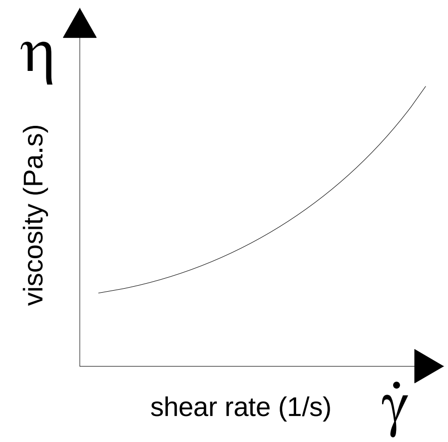

Figure 3.7: Viscosity curve for a shear- thickening behaviour.

Viscoplastic fluids

Viscoplastic materials are those having a yield stress which means that they will only flow when shear stress exceeds the yield point value. Theses samples behave as a solid below the yield stress value and as a liquid above that. The flow curve associated with this behaviour may be linear (Bingham behaviour) or non-linear. The Bingham model often presents materials with a yield value and a Newtonian behaviour- these samples are called Bingham plastic samples. For materials having a non-linear behaviour after a yield stress value they are generally best represented by the Herschel-Bulkley model- called viscoplastic materials (see 3.5.8.1). Figure 3.8 represent the flow curve behaviour of yield stress fluids.

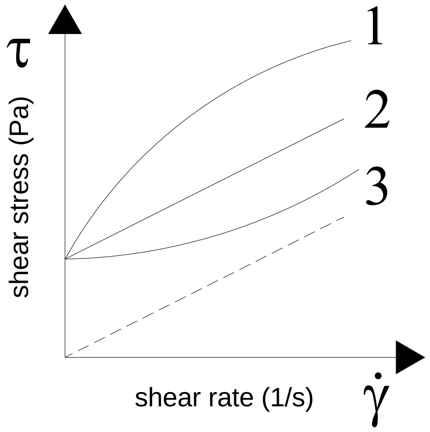

Figure 3.8: Flow curve behaviour of viscoplastic (yield stress) fluids: 1) shear thinning, 2) Bingham plastic, 3) shear thickening.

The viscoplastic behaviour is found in flocculated suspensions, like cement or hydraulic lime grouts, mortars and concretes (Wallevik J., 2009), (Hackley *et al.*, 2001), (Wallevik, 2009). They are dispersions where the dispersed particles form a network which is responsible for the yield point. The stronger the network, the larger the stress is necessary to overcome the internal structure. If the stress applied is lower than the yield value it will cause an elastic deformation of the sample and its shape will be recovered. On the contrary, if the shear stress is higher than the yield stress (also larger than the internal network force to resist breakage of structure) then it will result in a continuous flow.

Some controversy exists about the real existence of yield stress as a material property. Concerning the rate of shear which is of interest for cement based particle suspensions the yield value is real (Wallevik, 2009).

According to (Barnes *et al.*, 2001) many materials show not only a rheological behaviour. The changes of shear rate lead to different behaviour and normally three or four different regions may be observed, as can be seen in Figure 3.9.

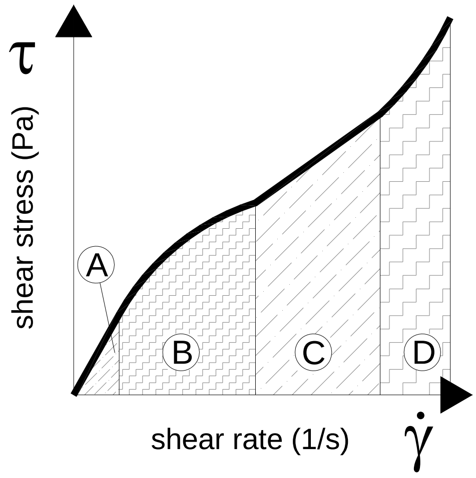

Figure 3.9: Different flow behaviour as a function of the applied shear rate in a material.

The figure shows the flow curve of a material in four different regions. The first one (A) refers to the first Newtonian region (showing a constant viscosity value- called the zero shear viscosity). In the second (B) it may be observed the shear-thinning behaviour where the viscosity decreases with an increase in shear rate. After that, a second Newtonian region may be detected at high shear rates (C). An increasing of shear rate may sometimes lead to a shear thickening region (D).

Viscoelastic fluid behaviour

While static measuring techniques are appropriate to characterize both time-dependent and -independent fluid behaviours, dynamic measuring techniques (oscillatory tests) or creep/recovery tests are necessary to characterize completely the viscoelastic fluid behaviour. In the absence of the time-dependent behaviour those types of materials show both elastic and viscous effects under appropriate circumstances, meaning that the shear stress depends both on the shear strain and shear rate (Nunes, 2008). Flocculated suspensions show viscoelastic behaviour at low strains (below the yield stress) (Struble *et al.*, 2001).

3.4.3 Non-Newtonian fluids – time dependent

The Non-Newtonian fluids- time dependent are the fluids for which the viscosity depends not only on the shear rate applied to the sample but also on the time for which the fluid has been subjected to shearing. This can be expressed as

$$\eta = \eta(\dot{\gamma}, t) \quad (3.7)$$

These types of materials change their structure with the time of shear at a constant shear rate, which affects the viscosity even for that situation. Time-dependent fluid behaviour may be further sub-divided into three categories (Wallevik, 2009):

- Thixotropic;
- Rheopectic;
- Anti-thixotropic.

Thixotropic

Thixotropic fluids have the ability to go from one structural state to another and back again to the initial state during a limited time. In principle all fluids having a microstructure can show thixotropic properties. Those materials show a shear-thinning and time dependent behaviour. Microstructure in this context means flocculated particles, but it could also be fibres arranging themselves favourably in the field of flow, droplets in an emulsion changing their solid geometry or molecules in a polymer solution regrouping from a tangled to a favourable system in the field of flow. Those structural systems variations present a large influence on material viscosity and elasticity. Maximum structure gives the maximum viscosity and elasticity and vice versa.

Cementitious materials generally show a thixotropic behaviour (Roussel *et al.*, 2007), (Fernàndez-Altable *et al.*, 2006), (Roussel, 2006). This behaviour originates from the microstructure of the matrix system, due to coagulation and flocculation of suspended particles and the time taken to change this microstructure. As the suspension is sheared, the weak physical bonds among particles are ruptured and the network among them breaks down into separate agglomerates, which can disintegrate further into smaller flocs whereas if the suspension is at rest the particles will start to coagulate or flocculate into agglomerates again.

The time for structural breakdown is normally considerably shorter than the time for the structural build-up because of the high shear rates during the breakdown. The structural build-up is controlled by diffusion and thermal motions, which leads to a long build-up time since they are very small compared to the shear rate.

During the breakdown the structure of molecules/particles starts to orient them in the line of flow which reduces viscosity. However, that shear-thinning behaviour does not occur simultaneously but under a measurable length of time causing the material to be time dependent (Barnes, 1997), (Barnes *et al.*, 2001), (Billberg, 2006).

One of the favourite ways of measuring thixotropy is to perform a loop test (Figure 3.10). A loop test means to linearly increase the shear rate (or shear stress) from zero to a maximum value and then to return at the same rate to zero. The test could then be repeated again and again until constant loop behaviour is seen. The area (A) between the up and down curve is a measure of material thixotropy. That area has the dimension of energy related to the volume of the sample sheared which indicates that energy is required to break down the thixotropic structure. However, this energy can not be characterised as a material parameter in the same way as the Herschel-Bulkley or Bingham parameters, as the area A depends on the shear history of the material and degree of dispersion (Wallevik J., 2003), (Mewis *et al.*, 2009), (Barnes, 1997).

No hysteresis loop is observed for time-independent fluids, that is, the enclosed area of the loop is zero. The following figure represents the thixotropic loop when the material is subjected to the loop test.

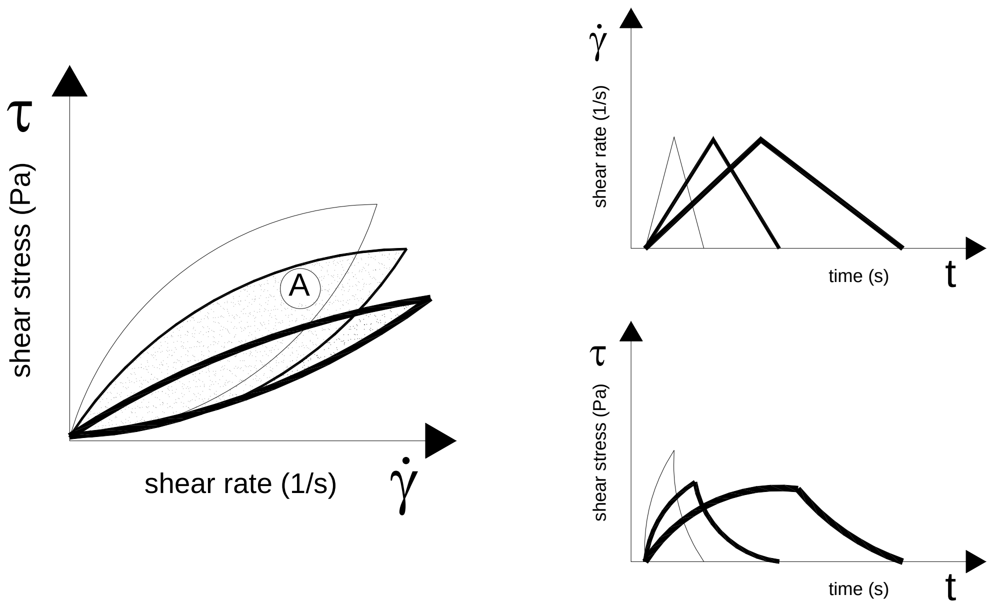

Figure 3.10: Representation of thixotropic material reaction due to loop change in the shear rate.

There are more ways to describe the behaviour of thixotropic materials (Barnes, 1997), (Barnes *et al.*, 2001), (Billberg, 2006). The most suitable way is to describe the material response in shear stress (τ) due to a shear rate ($\dot{\gamma}$). Figure 3.11 shows a schematic representation of a thixotropic material where the rate of shear is changed stepwise. In this case the material is subjected to rest and then to high shear rate $\dot{\gamma}_2$, where it will take long time to break down the agglomerate into particles and to reach an equilibrium stress- the steady state. After this, $\dot{\gamma}_2$ is instantly lowed to $\dot{\gamma}_1$. If $\dot{\gamma}_2 - \dot{\gamma}_1$ is small (going from a higher degree of dispersion to lower one) the time to reach the equilibrium is very small. On the contrary, if $\dot{\gamma}_2 - \dot{\gamma}_1$ is high instead of breakdown, agglomerates will rebuild again.

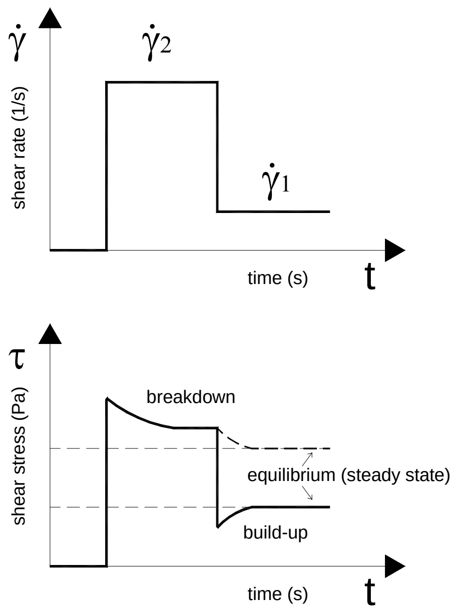

Figure 3.11: Schematic representation of a thixotropic material where the rate of shear is changed stepwise.

Other possible ways of studying material thixotropy using stepwise shear rate are represented in Figure 3.12 and 3.13.

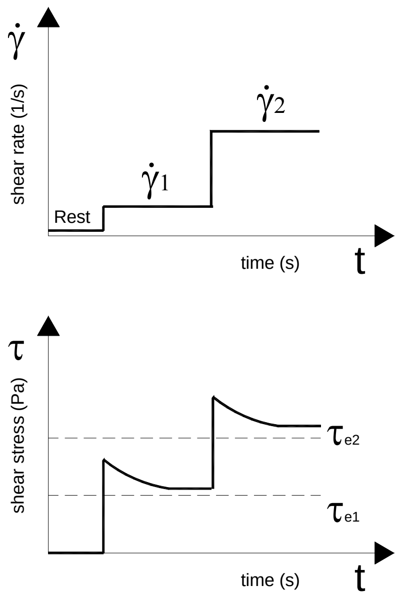

Figure 3.12: Stepwise increased shear rate with the material response as a reduced shear stress when the shear rate is constant (Billberg, 2006).

Figure 3.12 shows a step up test where shear rate is suddenly increased until a constant value. If the time at constant shear rate is sufficiently long in each step then the shear stress would reach the equilibrium values τ_{e1} and τ_{e2}, respectively. In this case the typical thixotropic behaviour is that the shear stress (or the viscosity since $\tau = \eta.\dot{\gamma}$) decreases at a constant shear rate.

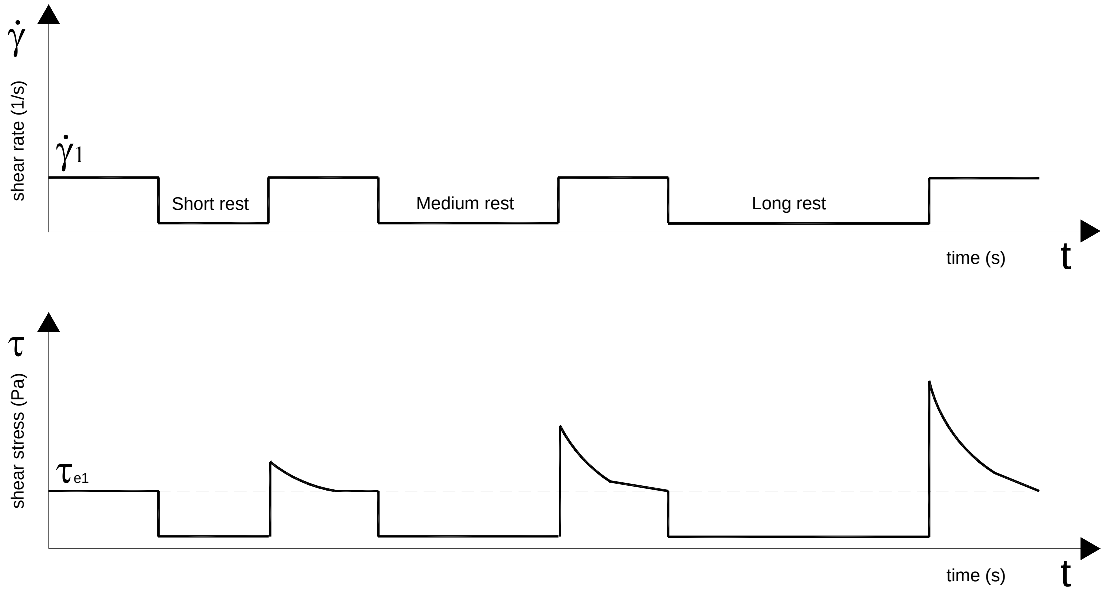

Figure 3.13: Structural build-up depending on resting time (Billberg, 2006).

The characterization of a thixotropic behaviour during a structural build-up at rest is shown in Figure 3.13. It is shown how the shear stress changes after a certain resting time, followed by a constant shear rate applied to the material. Since the shear stress reaches a high peak level each time (at $\dot{\gamma}_1$) after a longer resting time and then breaks down to τ_{e1} again it means that the level of structure in the material gets higher the longer it is allowed to rest.

Rheopectic

Rheopectic fluids show a structure breakdown at high shear rates similar to what occurs for thixotropic fluids. However, the main difference between those two materials is visible at low shear rates and at rest. A rheopectic fluid rebuilds its structure faster at low shear rate compared with at rest. For thixotropic materials is the opposite. Rheopectic fluids are not common (Wallevik, 2009). An example is the gypsum paste.

Anti thixotropic

This type of fluid has a structure that builds up under shear but breaks down at rest. It means that those fluids increase viscosity with time of shearing. There are relatively few antithixotropic fluids (Wallevik, 2009). Hysteresis effects are observed in the flow curve of the loop test, but in this case, it is inverted, as compared with a thixotropic material. An example of a thixotropic material is the rubber latex.

3.5 Factors influencing the rheology of hydraulic binder suspension

Hydraulic binder grouts are very complex and there are several factors that influence the rheology of fresh grout, such as: particle size distribution, volume fraction particles, particle form, chemical composition, solvability of ions from the surface, degree of flocculation (initially), agitation history, ability to disperse flocks, chemical admixtures, mineral admixtures, homogeneity/stability in regard of segregation (Wallevik, 2009).

The finer the cement or hydraulic lime, the more the colloidal forces will influence its movement and thereby the stronger will be the coagulation process. Together with the previous phenomenon, the finer the binder the more the number of particles will increase as well as the specific surface and each particle will absorb and bind more water molecules, so the water demand will be higher.

In cement grout, colloidal particles/forces and coagulation processes dominate the rheology as the major parts of the particles are of colloidal size. On the other hand, the rheology of fresh concrete is dominated by the volume of paste and friction in the particle system. The dispersion of flocculated cement aggregates will create some new surface area on some of the particles as a function of time (due to agitation, for instance). These areas will most likely obtain relatively high surface potential, which will increase coagulation process. The new area can consume or bind water and eventually chemical admixtures (Wallevik, 2009).

Traditionally, the particles that are influenced by the total potential energy effects are considered to be colloidal particles. A colloid is a system where one phase is dispersed in another phase for example between materials at different states of aggregation, i.e., gas/solid, liquid/solid, or between materials at the same state of aggregation such as oil/water. The dispersed phase of a colloid should by definition have at least one dimension (length, width, thickness) in the range 1 nm – 1 μm (Wallevik J., 2009). However, there is no clear distinction between the behaviour of particles with somewhat larger dimensions than those of the traditional colloidal particles.

According to (Wallevik J., 2009) it is demonstrated that cement particles as large as 40 μm in diameter seem to be able to “behave”, at least to some degree, as a colloid particle, somewhat controlled by the action of the total potential energy. Nevertheless, there are several forces acting on particles in a suspension.

3.5.1 Forces acting on particles suspended in a liquid

Rheology of suspensions depends on the forces acting on it and three kinds of forces coexist to various degrees in flowing suspensions (Barnes *et al.*, 2001):

a) Colloidal: those forces arise from interactions between the particles and can result in an overall repulsion or attraction between the particles, due to factors as van der Waals forces (attraction) and electrostatic charges (repulsion). If the net result of all the forces is an attraction, the particles tend to flocculate, whilst overall repulsion means that they remain separate (deflocculated). The previous phenomenon occurs when superplasticizers are added to cement grout, for example.
b) Brownian (thermal) randomising forces: they cause a rapid and random motion in very small particle (smaller than 1 μm). Brownian motion prevents settling of small particles even though they are denser than the suspending fluid.
c) Viscous forces: those forces acting on particles are proportional to the local velocity difference between the particle and the surrounding fluid. Thus, the suspension viscosity depends on the viscosity of the suspending medium.

3.5.2 Physical interactions

A fundamental theory in colloid science is the DLVO theory (developed by **D**erjaguin and **L**andau, **V**erwey and **O**verbeek 1941-1948). According to the DLVO, the stability of a colloidal suspension depends on the balance between the Van der Waals and the repulsive forces. It combines the effects of the van der Waals attraction and the electrostatic repulsion due to the so called double layer of counterions. Both the repulsion and the attraction forces increase quickly as the particles approach each other and it as been used to describe cementitious systems. This theory and relevant basic concepts are reviewed and applied to cement systems to determine the state of flocculation for cement pastes. Detailed information could be found in (Yanga *et al.*, 1997), (Vieira, 2008)**.**

Another potential energy able to play an important role in the dispersion of cement pastes is the steric effect. Steric effects arise from the fact that each atom within a molecule occupies a certain amount of space. If atoms are brought too close together, there is an associated cost in energy due to overlapping electron clouds (Pauli or Born repulsion), and this may affect the molecule's preferred shape (conformation) and reactivity. Steric hindrance or steric resistance occurs when the size of groups within a molecule prevents chemical reactions that are observed in related smaller molecules. Although steric hindrance is sometimes a problem, it can also be a very useful tool and is often exploited by chemists to change the reactivity pattern of a molecule by stopping unwanted side-reactions. It plays an important role in the dispersion of cement pastes in the presence of superplasticizer (Vieira, 2008).

3.5.3 Influence of solid particles in rheology of suspensions

For diluted suspensions, an increase of the solid volume fraction (ϕ - the ratio between the solid phase volume and the mixture volume) increase the viscosity of the suspension (η) (Barnes *et al.*, 2001). Einstein showed that single particles increased the viscosity of a Newtonian liquid as a simple function of their phase volume according to the formula:

$$\eta = \eta_s (1 + 2.5\phi) \qquad (3.8)$$

where η_s is the viscosity of the suspending medium. In equation 3.8 there is no effect of particle size, nor of particle position, because the theory neglects the effects of other particles. However, this equation is not indicated for fluids with higher concentration of solid particles such as grouts, mortars or concretes.

The influence of particle concentration on the viscosity of the concentrated suspensions is best determined in relation to the maximum packing fraction (ϕ_m). ϕ_m is the solid volume fraction that correspond to the maximum volume of solid particles able to be added to a certain volume of fluid. As more and more particles are added, there will a time when flow will be impossible and the viscosity tends to infinity. Maximum packing fraction depends on the arrangement of the particles. Table 3.2 presents some examples.

Table 3.2: Maximum packing fraction of various arrangements of monodisperse spheres (Barnes *et al.*, 2001).

Arrangement	**Maximum packing fraction**
Simple cubic	0.52
Minimum thermodynamically stable configuration	0.548
Hexagonally packed sheets just touching	0.605
Random close packing	0.637
Body-centred cubic packing	0.68
Face- centred cubic/hexagonal close packed	0.74

The maximum packing fraction is also very sensitive to particle- size distribution and particle shape. Broader particle- size distributions have higher values of ϕ_m because the smaller particles fit into the gaps between the bigger ones. On the other hand, nonspherical particles lead to poorer space-filling and hence lower ϕ_m. Low ϕ_m could also occur when particle flocculation happens, since the flocs themselves are not close-packed. From the previous statements it could be noted that the ratio ϕ / ϕ_m is a relevant normalized concentration.

Thus, developments to the Einstein equation have been made (Barnes *et al.*, 2001). One of the most adopted is the Krieger-Dougherty equation, to describe concentrated suspensions:

$$\eta = \eta_s (1 + \phi / \phi_m)^{-A\phi_m} \tag{3.9}$$

where A is the intrinsic viscosity of the medium.

Concentrated suspensions (or “hard” suspensions) are obtained when particle interactions play a major role in the suspension behaviour. Each particle is submitted to various forces such as colloidal, Brownian, viscous forces and contacts (lubrication, friction, collision). Thus, different rheological behaviour of suspensions will be obtained in each regime.

3.5.4 Rheophysical regimes of a suspension

Supposing that suspension parameters, such as ionic strength, particle diameter and shape, flow geometry, temperature, friction coefficient, viscosity of the interstitial fluid, fluid and particle density, external force and boundary conditions, are constant except ϕ and $\dot{\gamma}$. Coussout *et al.* in (Coussot *et al.*, 1999) provided a simplified conceptual diagram of predominant interactions within flowing concentrated suspensions (mainly under simple shear) as a function of shear rate and solid fraction (ϕ) (Figure 3.14). They also provided some elements concerning the typical rheological behaviour of suspensions in each regime.

However, for higher particle diameter (large grains), the curves corresponding to the transition zone from regime (A) or (C) toward regime (B) would be displaced toward infinitely small shear rates, which means that hydrodynamic effects will prevail.

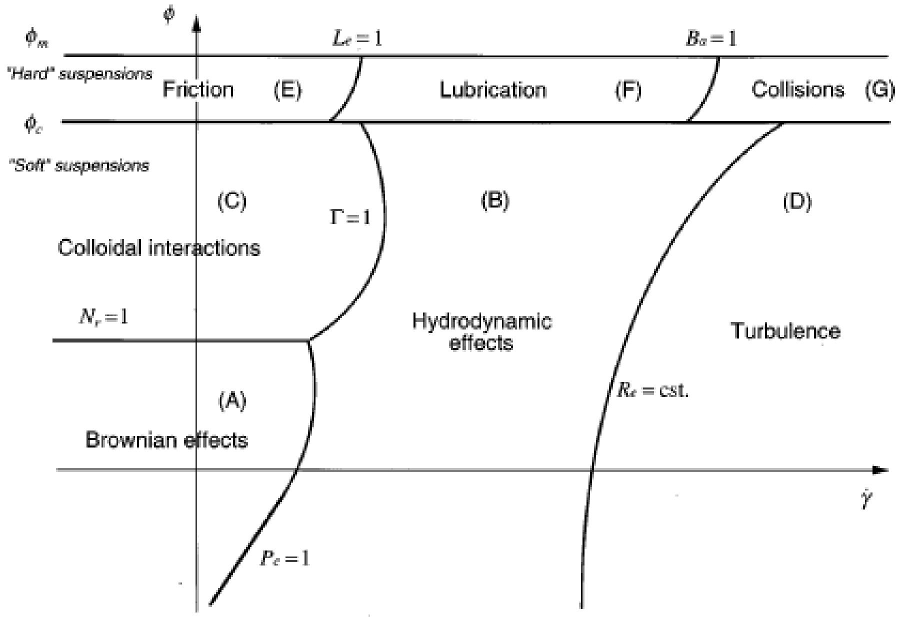

Figure 3.14: Classification of the rheophysical regimes of a suspension as a function of shear rate and solid fraction on a logarithmic scale (Coussot *et al.*, 1999).

The present scale ($\phi, \dot{\gamma}$) is used for the presentation of results since it contains two parameters providing rapid basic information concerning the density of the suspension and the flow intensity.

When a suspension is at rest (or under very low shear rates) Brownian or colloidal forces dominate, for a given solids volume concentration below a critical value (ϕ_c). A critical solids volume concentration is obtained when a network of contacting particles exists and friction forces become dominant. When the particle interference makes flow impossible and the viscosity approaches infinity maximum packing fraction (ϕ_m) is obtained.

Suspensions with low solids volume fractions (or "soft" suspensions) and low shear rates exhibit shear-thinning behaviour since they are dominated by Brownian motion. Sufficiently increasing the shear rate leads to turbulent flow.
As the concentration is increased, colloidal interactions dominate at low shear rates and the suspension exhibits yield stress. In addiction it is viscoelastic and thixotropic (see Figure 3.14). If shear rate is increased from friction zone, the imposed velocity gradient imposes an

orientation of the particle structure, a thin layer of fluid exists between particles which lubricate the contacts, and then the viscosity is lowered (shear-thinning behaviour).

It is expected that a clearer distinction of significant interactions in different regimes constitutes a necessary first stage in order to understand these systems better in terms of modeling the rheological behaviour. It could be highlighted the influence of shear rate range in the type of particle interactions obtained.

3.5.5 Rheophysical regimes of a cement suspension

Cement particles are less sensitive to Brownian motion. They present a broader particle- size distribution, so it means that probably the particles are distributed between several regimes (Coussot *et al.*, 1999b). Thus, cement suspensions are somewhere between the regimes (C) and (E) or (B) and (F) for shear rate increase (Figure 3.15). These interactions lead to a more complex behaviour of cement systems flow.

The flocculated state of cement particles, namely colloidal ones, is responsible for plastic behaviour, with the yield stress reflecting the forces holding particles together. Often this breakdown is not complete at the yield stress point, so the suspension is still somewhat flocculated even though it flows and this remaining flocculation is progressively disrupted as the shear rate is increased (Nunes, 2008), (Struble *et al.*, 2001), (Rosquoët *et al.*, 2003). This increase of flow velocity will also lead to an increase of overall repulsion, due to fluid pressure, meaning that particles remain separate (deflocculated) and without any friction.

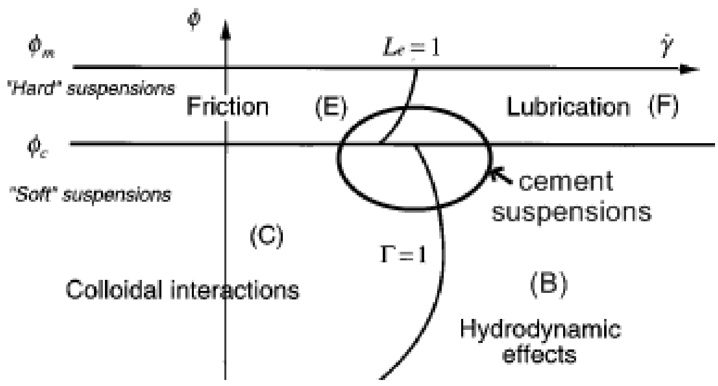

Figure 3.15: Rheophysical regimes of a cement suspension as a function of shear rate and solid fraction on a logarithmic scale.

Like changes in shear rate modify the mix rheology, water will also modify it. The addition of water will increase the average distance between particles and the possibility for coagulation process decrease. Because of the increased water content in the suspension, the particles or flocks of particles can more easily move around each other and viscosity will reduce significantly. Since the distance between particles is greater, the necessary stress to make an initial moment is lower, which means that yield stress value will reduce when water is added (Wallevik, 2009).

Shear-thinning or shear thickening behaviours may be observed depending on the analysed range of shear rate (Atzeni *et al.*, 1985), (Roussel, 2005). However, the apparent shear thickening behaviour of cement grout may also be an artefact from the experimental set-up, caused by a lack of steady state during the rheological measurements. This means that the relaxation period needed to obtain steady-state flow should be taken into account in the selection of measuring procedures (Geiker *et al.*, 2002), (Bager *et al.*, 2001).

3.5.6 Range of interest of shear rate

According to (Rosquoët *et al.*, 2003), (Wallevik, 2009) it is possible (but difficult) to estimate the range of interest of shear rate concerning the field of application of grouts, mortars and concretes. For concrete, the estimated shear rate that occurs during placing is approximately in the range of $3s^{-1}$ to $20s^{-1}$. For self-compacting concrete (SCC) it can be much less than $1s^{-1}$ which is so low that thixotropic phenomena will most likely start to thicken the concrete and thereby slow the speed further down until it stops. Wallevik also says that the upper limit of the shear rate range which is of interest to cement grout is as high as 60 to 80 s^{-1}.

A very high shear rate (from some several hundreds s^{-1}) will generate a different structure in the cement based particle suspension due to, for example, dilatancy. The suspension will create many layers with a high concentration of particles with a layer between them, containing liquid. Thus, the flow character could be completely different from what will occur during placing.

The following figure (Figure 3.16) presents the range of shear rate according to (Saak, 2000).

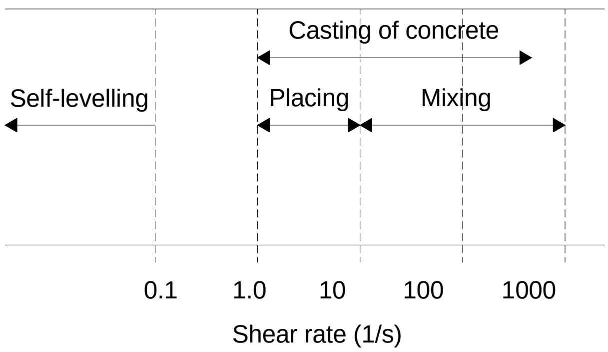

Figure 3.16: Range of shear rate used in rheological measurements according to (Saak, 2000).

The fields of use for hydraulic binder grouts are many: filling, sealing, consolidation, among others. During grout/concrete injection the slippage layer nearest to the wall of the pipe is usually a very dilute binder suspension relative do the paste in the core. When pumping, if the yield stress value is high, the grout/concrete core will be transported as a plug without shearing. If the grout/concrete is very fluid and the yield stress is significantly lower than necessary to maintain plug, shearing in the cross section of the pipe will occur. As the grout/concrete has much higher viscosity than the slippage layer, the necessary pump pressure to maintain movement will be much higher. This previous phenomenon occurs inside a porous media (like masonry) being injected by grout. The water absorption capacity of the porous media will reduce the slippage layer and an increased pump pressure will be necessary. So, the rheology of slippage layer depends on the ability of the grout/concrete to bleed under pressure.

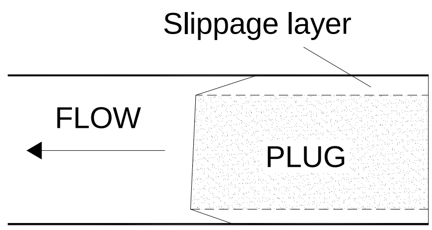

Figure 3.17: Slip layer next to the inner surface of the pipe during pumping of grout.

The type of grout/concrete application also affects the range of water/binder ratio (w/b) to adopt in the composition. Hydraulic grouts for the repair and consolidation of masonry structures have w/b ratios in the range 0.5–1.5 (Miltiadou, 1990). For the coating of prestressed cables, the w/b ratios found in the literature range between 0.35 and 0.42. For sealing cement grouts, w/b ratios are similar to those used for the repair and consolidation of masonry and range between 0.5 and 1. Cement grouts for soil or rock injection are very fluid hydraulic binders with w/b ratios between 1 and 2 (Figure 3.18).

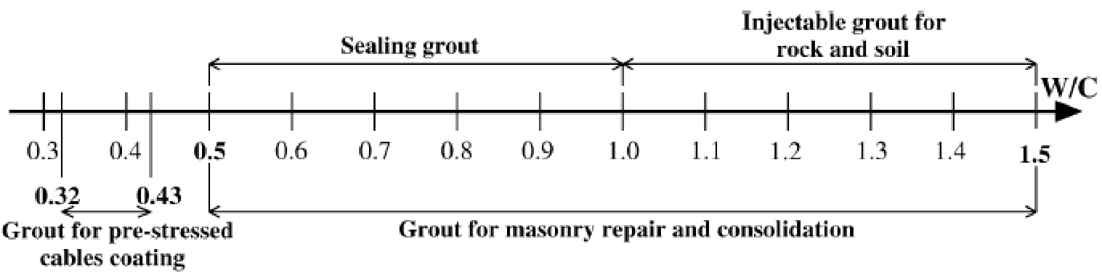

Figure 3.18: Scheme of different fields of use of cement grout (Rosquoët *et al.*, 2003).

Generally it is difficult to obtain accurate data regarding the rheology of fresh cement based particle suspension. A great effort has been spent to obtain accurate and repeatable data on the rheological material parameters since they may depend on the technique or instrument used. Now, the determination of rheological material factors/parameters of interest may be determined as accurately as most major parameters in concrete technology such as strength, elasticity or shrinkage (Wallevik, 2009).

3.5.7 Surface and colloid chemistry of thixotropic suspensions

Nowadays, there is quite a general agreement in the scientific community that thixotropy should be defined as: the continuous decrease of viscosity with time when flow is applied to a sample that has been previously at rest and the subsequent recovery of viscosity in time when the flow is discontinued (Mewis *et al.*, 2009).

The basic items for thixotropic definition normally used are that:
i) It is based on viscosity;
ii) It implies a time-dependent decrease of the viscosity induced by flow;
iii) The effect is reversible when the flow is decreased.

Many mechanisms behind thixotropy of particle suspensions can be found in the colloidal domain. Thus, the reason why the size aspect is so important is the high ratio between the areas of the dispersed phase relative to its volume, i.e., the specific surface area. The surface characteristics of this interface affect the whole physical properties of the system considerably since the surface may control interactions between particles-particles or particles-dispersion medium in a colloidal system.

Interparticle forces originate from the interatomic and intermolecular forces on the particle surface. These forces are described in terms of intermolecular energy potential. The derivate of potential energy function may represent the attraction forces (positive tangent- van der Waal forces) or the repulsion forces (negative tangent-Born forces). At small distances between molecules Born repulsion forces dominates while at long intermolecular distances van der Waals attraction dominates (Shaw, 1992).

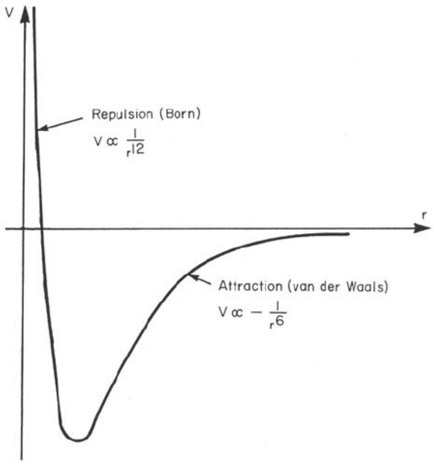

Figure 3.19: Forces between atoms or molecules expressed as a pair potential energy (Cheng, 1987).

According to (Barnes *et al.*, 2001) overall repulsion between the particles of a suspension is created if the particles carry electrostatic charges of the same sign. Thus, particles take up positions as far from one another as possible. Flow will only occur if particles are forced to leave its equilibrium position and are induced to move against the electric fields of neighbouring particles into nearby gaps in the imperfect network. Repulsion can also arise from the entropic forces caused by the interaction of the chains of any polymer adsorbed onto the particle surfaces. This could happen with the addition of dispersing admixtures in cement grout, like superplasticizers, that reduce yield stress value.

The complex rheological behaviour of thixotropic materials is most often the result of relatively weak attractive forces between the particles. They will cause the formation of flocs, which normally evolve into a space-filling particulate network. The interparticle bonds are, however, weak enough to be broken by the mechanical stresses that occur during flow. The result is that during flow the network breaks down in separate flocs, which decrease further in size when the strain rate is increased. When flocculated suspensions are sheared, the flocs rotate, possibly deform and if the applied stress is high enough, begin to break down to the primary particles.

Reducing and stopping the shear rate can cause a growth of the flocs and will allow the particulate network to rebuild, since particles will start to coagulate and flocculate into agglomerates. The driving force to rebuild the flocs is Brownian motion and since this increase with decrease in particle size, the rate of thixotropic change is a function of particle size. Thus, it is expected that systems of large particles recover their viscosity slower than systems of small particles. In the same way, large- particle suspensions will breakdown faster under shearing. These considerations are important in the design of thixotropic products (Barnes *et al.*, 2001), (Mewis *et al.*, 2009).

The influence of particle size on type of force acting on the particles is great as it could be seen from the previous sentences. Surface forces such as van der Waal forces, electrostatic forces and Brownian motion can only dominate over gravitational and inertial forces if the particle size is small enough. Table 3.3 presents different particle size and the energy associated to the type of interaction present.

Table 3.3: Energy for particles in a suspension depending on the interaction of different type of forces (Pugh *et al.*, 1994).

	Energy (in units of kT) for particles of given size		
Type of interaction	**0.1μm**	**1μm**	**10μm**
van der Waals attraction	10	100	1000
Electrostatic repulsion	0-100	0-1000	0-10000
Brownian motion	1	1	1
Kinetic energy of sedimentation	10^{-13}	10^{-6}	10
Kinetic energy of stirring	1	1000	10^{6}

As it can be observed, surface forces such as van der Waal forces, electrostatic forces and Brownian motion can only dominate in the range of 0,1 μm. For bigger particles, around 1-10 μm, the gravitational and inertial forces are important and the forces arising from stirring and mixing can give the system enough energy to separate flocculated particles.

3.5.8 Measurement techniques

According to Hackley and Ferraris in (Hackley *et al.*, 2001) , (Hackley *et al.*, 2001b), there are two common methods used for rheometric measurements on fluid systems: capillary (or tube) and rotational.

In capillary methods the test fluid is made to flow through a narrow tube as a result of hydrostatic or applied pressure. Those measurements are considered the most precise way of determining the viscosity of Newtonian and some non-Newtonian viscous fluids, and are generally simpler in design and less expensive relative to rotational instruments. The basis for the capillary method is the Poiseuille's Law, which relates the rate of flow through a capillary to the viscosity of the liquid.

In rotational methods the test fluid is continuously sheared between two surfaces, one or both of which are rotating. These devices are able to shear the sample for an unlimited period of time, permitting transient behaviour to be monitored or an equilibrium state to be achieved, under controlled rheometric conditions (Hackley *et al.*, 2001). Rotational methods can also incorporate oscillatory and normal stress tests for characterizing the viscoelastic properties of samples. It could be said that rotational methods are better suited for the measurement of concentrated suspensions, gels and pastes, but are generally less precise as compared to capillary methods. This type of measurements fall into one of two categories: rate-controlled or stress-controlled.

In rate-controlled measurements, a constant rotation speed is maintained and the resulting torque generated by the sample is determined using a suitable stress-sensing device, such as a torsion spring or strain gauge. In stress-controlled measurements, a constant torque is applied to the measuring tool in order to generate rotation and the resulting rotation speed is then determined. If a well-defined tool geometry is used, the rotation speed can be converted into a corresponding shear rate. Some commercial instruments have the capability of operating in either stress-controlled or rate-controlled modes.

Measurement instruments can be one of two types: a viscometer or a rheometer. A viscometer is an instrument that principally allows measuring viscosity; it is simpler in design and less expensive than rheometers. In opposition, a rheometer is an instrument used for the measurement of rheological properties over a varied and extended range of conditions.

Two devices were used in this research work for grouts:

- Viscometer Brookfield;
- Rotational rheometer from Bohlin Instruments.

Those instruments will be briefly described in the following point.

Viscometer Brookfield

The least expensive commercial variant of the controlled-rate rotational viscometer is commonly referred to as a "Brookfield type" viscometer. The model used was the Brookfield viscometer LV DV-II+PRO (Figure 3.20). This device measures fluid viscosity at fixed rotation speeds by driving a measurement tool ("spindle"), immersed in the test fluid (the grout), through a calibrated torsion spring. Due to viscosity of fluid the spindle causes the spring to deflect and this deflection is correlated with torque. Conversion factors are needed to calculate viscosity from the measured torque and are typically pre-calibrated for specific tool and container geometries. Knowing the tool geometry, the shape, the size of the sample container and the rotation speed, it is possible to calculate the shear rate.

For Newtonian fluids the torque is proportional to the product of viscosity and rotational speed, but this proportionality is lost in the case of a non-Newtonian fluid. Because these instruments are robust and fairly simple to use, they have found wide application in industry, but they offer limited capabilities and precision for research-oriented applications (Hackley *et al.*, 2001).

Figure 3.20: Brookfield viscometer LV DV-II+PRO (Bras *et al.*, 2009).

Rotational rheometer from Bohlin Instruments

Rotational rheometer are of higher-precision comparing with viscometers. The test fluid is sheared between rotating cylinders, cones or plates, under controlled-stress or controlled-rate conditions. It is also a continuously-variable shear instruments. Some instruments have the capability of operating in stress-controlled as well in shear-rate controlled modes. Instruments producing oscillatory strains are available and a few commercial systems permit measurement of the normal stress.

In rate-controlled measurements, a constant rotation speed is maintained and the resulting torque generated by the sample is determined using a suitable stress-sensing device, such as a torsion spring or strain gauge. The rotation speed is converted into a corresponding shear rate, based on the geometry of the measuring tool. In stress-controlled measurements, a constant torque is applied to the measuring tool in order to generate rotation, and the resulting rotation speed is then determined.

The basic rotational system consists of four parts (Hackley *et al.*, 2001):

i) A measurement tool with a well-defined geometry;

ii) A device to apply a constant torque or rotation speed to the tool over a wide range of shear stress or shear rate values;

iii) A device to determine the stress or shear rate response;

iv) Some means of temperature control for the test fluid and tool.

Most rheometers are based on the relative rotation about a common axis of one of three tool geometries: concentric cylinder, cone and plate or parallel plates (Figure 3.21).

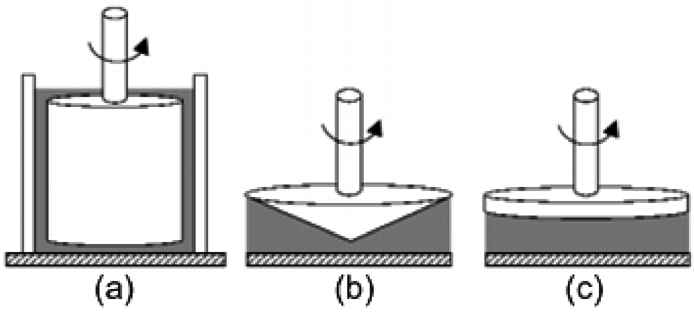

Figure 3.21: Schematic diagram of basic toll geometries for the rotational rheometer: (a) concentric cylinder, (b) cone and plate, (c) parallel plate (Hackley *et al.*, 2001).

The rotational rheometer adopted in the current work was the Bohlin Gemini HR^{nano} rotational rheometer, in a parallel plate (plate/plate) geometry, from Bohlin Instruments (Figure 3.22).

Figure 3.22: Main parts in the rotational rheometer used in the present work: (a) gap size indicator; (b) device to determine the shear rate (or stress) response and a device to apply a constant torque (or rotation speed) to the tool, respectively; (c) measurement tool; (d) thermostatic bath with temperature control.

The parallel plate geometry can be considered a simplified version of the cone and plate, having an angle of 0°. The test fluid is constrained in the narrow gap between the two surfaces. Cone and plate and parallel plate measurement tools are most often used for highly viscous pastes, gels and concentrated suspensions (Hackley *et al.*, 2001). Thus, it was selected in the research work.

3.5.8.1 Shear rate dependence

To describe the shear-thinning behaviour of hydraulic lime or cement based grouts several models could be adopted (Table 3.4):

Table 3.4: Rheological models (Barnes *et al.*, 2001).

Rheological models	Equation
Bingham	$\tau = \tau_0 + \eta_p \times \dot{\gamma}$
Modified Bingham	$\tau = \tau_0 + \eta_p \times \dot{\gamma} + c \times \dot{\gamma}^2$
Herschel-Bulkley	$\tau = \tau_0 + A\,\dot{\gamma}^n$
Casson	$\tau = \tau_0 + \eta_\infty \dot{\gamma} + 2 \times (\sqrt{\tau_0 \eta_\infty})\sqrt{\dot{\gamma}}$
Sisko	$\eta = \eta_\infty + K_2 \dot{\gamma}^{n-1}$

Where *A* and *n* are characteristic parameters describing the shear rate dependency of the material tested, η_∞ is the second Newtonian region and K_2 is called the consistency.

Bingham model is the most used for rheology characterization. However, it is now admitted that this model lacks physical sense and, contrary to Herschel-Bulkley, it is incapable of representing data within a shear rate range of decades.

Figure 3.23 shows flow-curve for Herschel-Bulkley fluids relative to Bingham fluids. When *n>1* the equation signifies shear-thickening behaviour and *n<1* means shear-thinning behaviour.

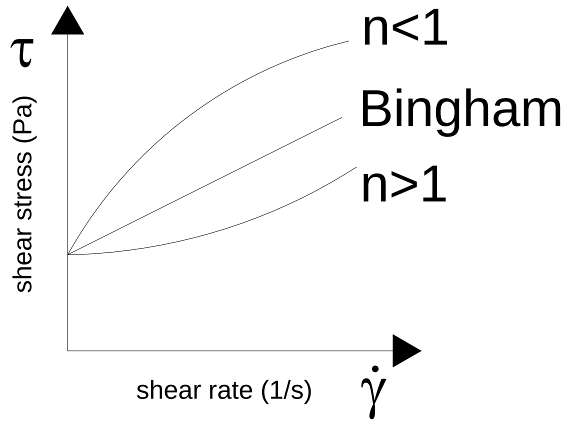

Figure 3.23: Flow curve for a Herschel- Bulkley and Bingham fluid.

More detailed information about the rheological measurements and techniques could be found in the following Chapters.

3.5.8.2 Shear rate and time dependence

According to Roussel in (Roussel, 2006), (Roussel, 2005) as long as steady state flow is reached, the behaviour of cement grouts may be described using a yield stress model such as the Bingham or Hershel Bulkley models. However, between two successive steady states, there is a transient regime, during which a yield stress model is not sufficient to describe the observed behaviour.

The theoretical line expected from a simple yield stress fluid (bold line) and experimental lines (dashed line) of flow behaviour is represented in Figure 3.24 and 3.25.

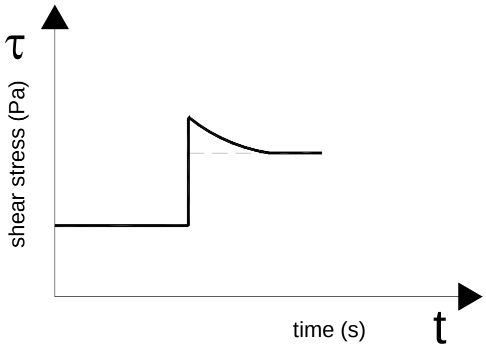

Figure 3.24: Examples of transient behaviour: rotating speed increase.

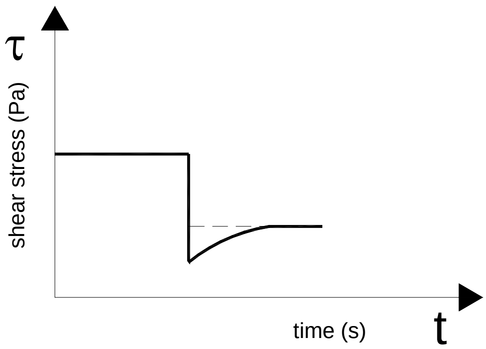

Figure 3.25: Examples of transient behaviour: rotating speed decrease.

The difference is due to the thixotropic behaviour of the tested sample that creates a delay in the material answer. That delay, in the case of cement pastes, can be correlated to the applied shear rate and to the recent flow history of the material.

For a low shear rate application in a material that was at rest before it will be detected behaviour similar to Figure 3.26.

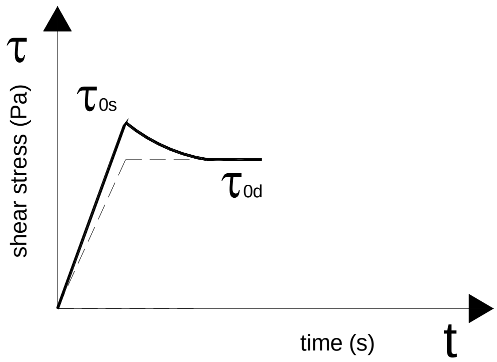

Figure 3.26: Rotating speed increase after a resting period.

After a linear increase due to the elastic part of the behaviour, a static yield stress τ_{0s} can be measured followed by a dynamic yield stress τ_{0d} corresponding to steady state The static yield stress τ_{0s} increases with the resting time before the test but the τ_{0d} does not depend on the material flow history (Roussel, 2006).

At low shear rates a suspension may be very slow to reach equilibrium. In the case of cement paste, it may not be possible to wait for such a long time, because of other time dependent processes that are going on, like cement hydration, as the hydration process starts as soon as cement and water are mixed together. To be able to compare measurements in common references and minimize the influence of past shear history (due to mixing and handling of sample) it is advisable to pre-condition the sample before the start of the viscometry test (Saak, 2000).

This means that transient flow effects are important, as shown for example by Otsubo *et al.*. (Otsubo *et al.*, 1980) who found that the flow properties of homogeneous fresh hydraulic based grouts (like cement) continuously evolve in time. Banfill and Saunders (Banfill *et al.*, 1981) and Lapasin *et al.* (Lapasin *et al.*, 1983) demonstrated that for a constant shear rate the first regime is dominated by a de-flocculation phenomenon under constant shear rate (thixotropic behaviour). Once this phenomenon has reached some equilibrium, the behaviour keeps on evolving because of hydration process.

There are some models to describe the competition between breakdown and structural build-up that probably could be adopted in grout or concrete characterization. Cheng and Evans in (Cheng *et al.*, 1965) proposed a general mathematical form of the equation of state of a thixotropic material. These authors assumed the existence of a shear and time dependent structure. The model proposed consists in a constitutive equation, which links the shear stress to the shear rate by means of a shear rate viscosity function. This equation is obtained from data corresponding to equilibrium states. In a subsequent phase of the model application, it is imposed the viscosity also depends on a structural parameter (λ) which is related to the flocculation level inside the material. Therefore, the general expression for the constitutive equation of the material will be,

$$\tau = \eta(\lambda, \dot{\gamma})\,\dot{\gamma} \tag{3.10}$$

To the constitutive equation it is added a kinetic equation, which expresses the time evolution of the microstructure from a non-equilibrium state to an equilibrium one:

$$\frac{d\lambda}{dt} = f(\dot{\gamma}, \lambda) - g(\dot{\gamma}, \lambda) \tag{3.11}$$

where the function *f* express the build-up and *g* express the breakdown of the dynamical structure during the transient state.

Coussot *et al.* in (Coussot *et al.*, 2002) proposed a simple model that describes the competition between aging and rejuvenation. They suppose that the degree of jamming can be described by a single parameter λ, where λ represents the degree of flocculation. For an aging system like cement paste, at rest λ increases at a constant rate $\frac{1}{\theta}$ where θ is the characteristic time of evolution of the structure. The rate of decrease of λ under shear is assumed proportional to both the shear rate and the degree of jamming, leading to an evolution equation for λ:

$$\frac{d\lambda}{dt} = \frac{1}{\theta} - \alpha\lambda\dot{\gamma} \tag{3.12}$$

where α and θ are material parameters, $\dot{\gamma}$ the shear rate and *t* the time. To relate flow and structure, they adopted a general viscosity function:

$$\eta = \eta_0(1 + \lambda^n) \tag{3.13}$$

where η_0 is an asymptotic value of viscosity that is reached when structure is entirely destroyed. According to Roussel in (Roussel, 2006), this previous model has been validated by local and macroscopic comparison between MRI (Magnetic Resonance Imaging) experiments carried out on Bentonite suspensions and simulations. Roussel also suggest a simple thixotropic model for fresh concrete based on the principles described before:

$$\tau = (1 + \lambda)\tau_0 + k\dot{\gamma}^n \tag{3.14}$$

$$\frac{d\lambda}{dt} = \frac{1}{T\lambda^m} - \alpha\lambda\dot{\gamma} \tag{3.15}$$

where the λ is again the flocculation rate, *T*, *m* and α are thixotropic parameters.

Just after mixing $\lambda = 0$ and this means that $\lambda\tau_0 = 0$. With the successive steps in the process (rest phase, re-mixing phase, pumping phase, among others) λ will evolve to a positive value according to (3.15) and an apparent yield stress greater than τ_0 will appear.

Supposing that a Bingham behaviour is valid for the steady state characterization of fresh concrete then *n=1* and $k = \eta_p$. Since yield stress at rest increase in a linear way with time for this material then *m=0*.

$$\tau = (1+\lambda)\tau_0 + \eta_p \dot{\gamma}^n \tag{3.16}$$

$$\frac{d\lambda}{dt} = \frac{1}{T} - \alpha\lambda\dot{\gamma} \tag{3.17}$$

where the first parcel in (3.17) refers to the flocculation state and the second to the breakdown of the structure.

$$\text{At rest} \quad \begin{cases} \dot{\gamma} = 0 \\ \alpha\lambda\dot{\gamma} = 0 \end{cases} \Rightarrow \tau_0(t) = \tau_0 + \tau_0\frac{t}{T} = \tau_0 + A_{thix}t \tag{3.18}$$

where A_{thix} is the rate of flocculation in Pa/s.

Hattori and Izumi developed the "Coagulation Rate Theory" which can be used as a mathematical tool to describe thixotropy (Wallevik, 2009). The theory is general and probably could be used on all kinds of multiple-phase suspension, in particular (two-phase) suspensions like grouts. The theory was applied and developed in the numerical simulations carried out by Wallevik (Wallevik, 2009). According to this theory, the viscosity of a suspension as a function of time is given by

$$\eta = B_3 J_t^{2/3} = B_3\left[n_3\frac{U_0(\dot{\gamma}Ht^2+1)+Ht}{(Ht+1)(\dot{\gamma}t+1)}\right]^{2/3} \tag{3.19}$$

where B_3 is the friction coefficient between particles; n_3 is the number of particles; U_0 is the initial degree of dispersion and *H* is the coagulation rate constant (Wallevik, 2009).

3.5.9 Thixotropy of hydraulic binder grouts - engineering consequences

Thixotropic materials, such as cement or hydraulic lime grouts, show a shear-thinning and time dependent behaviour. This behaviour originates from the microstructure of the matrix system, due to coagulation and flocculation of suspended particles and the time taken to change this microstructure. As the suspension is sheared, the weak physical bonds among particles are broken and the network among them breaks down into separate agglomerates, which can disintegrate further into smaller flocs (structural breakdown). If the suspension is at rest the particles will start to coagulate or flocculate into agglomerates again (structural build-up).

A rheological material model is presented by J. Wallevik to describe time dependent behaviour of the cement paste with different types of admixtures. It is named the Particle Flow Interaction Theory, or the PFI-theory. This behaviour is described for cement paste (Wallevik J., 2009) (see Figure 3.27).

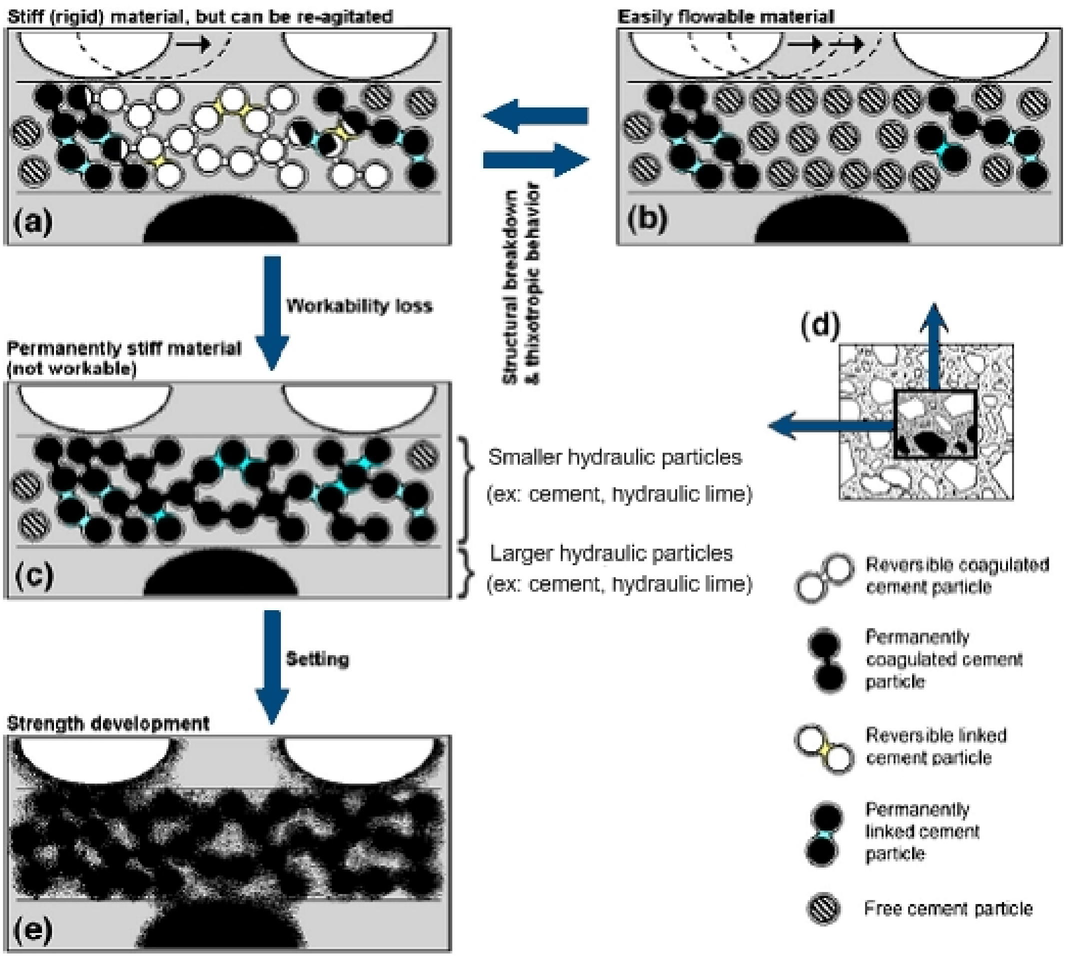

Figure 3.27: Visual summary of the most important aspects of the PFI-theory, based on (Wallevik J., 2009).

In Figure 3.27 (a) it is represented the cement paste after some resting (10 minutes). The connections between the cement particles are of both reversible and permanent nature and some cement particles are still free. With some agitation the particles with reversible connections become free and the others remain together (b). It is possible to return from state (a) to (b) for short period of time.

For a longer resting time, more and more cement particles would become permanently connected, which leads to workability loss according to the PFI-theory (Wallevik J., 2009). In (c), most of the cement particles have been permanently connected. Therefore, the cement paste cannot flow, not even with the effort of re-agitation. The cement pastes shown in Figure 3.27 (a) and (c) have a similar consistency. Their difference consists in that the former can be re-agitated to make it flowable, while the latter not. In (e), strength development occurs due to setting.

Like for concrete, a good grout is the one that satisfies certain requirements according to the specific application and several questions should be answered first, before take a decision, such as:

Are they for coating of prestressed cables?
Are they for repair and masonry consolidation?
Are they for sealing?
or for injection in soil or rocks?
Does the grout need to be self levelling?
Does it need to be pumped?
Is the grout for application in wall or slab?

From a rheological point of view, the goal is to design a homogeneous grout that is easily placed at the lowest possible cost. There are many ways to apply rheology in grouts and concrete technology. One can use it during mix proportioning or mix design, for raw materials evaluation or in quality control at building site. Steady state and transient state material's characterization enables to get that information and to make a decision about. It could be also said that the development of thixotropic grouts should focus on state (a) and (b).

Barnes in its state of art concerning thixotropy (Barnes, 1997) mentioned the easy way to characterise the flow of thixotropic materials in mixes if it is assumed that the mixer behaved in the same way as a viscometer, running at the same shear rate as the average shear rate in the mixer. The important issue is to equal shear rate mixer to the shear rate of viscometer in order to get a good rheological characterization.

As a first step, the ability of a grout to perform as desired is commonly assessed using bulk flow properties measured in the laboratory, such as its yield stress and plastic viscosity, since they may also be used as a prediction factor of whether or not a grout will be able to be pumped, among others. It is clear, however, that additional characterization of the flow and injection processes are needed, due to the complex composition of grouts and the complex interaction of flows and flow geometries which may involve in time, as it was explained before.

(Roussel, 2006), (Billberg, 2006) statements that rheological phenomenon such as thixotropy of SCC plays an important role in the values of lateral stresses in the formwork when casting. During placing, the concrete behaves as a fluid but, if cast slowly enough or if at rest, it builds up an internal structure and has the ability to withstand the load from concrete cast above it without increasing the lateral stress against the formwork. That lateral stress does not reach the hydrostatic pressure when concrete is cast from the top of the formwork slowly enough to flocculate and withstand the load of concrete cast above it.

Among that, because the material builds up an internal structure, the lateral stress decreases quickly in the part of concrete that is at rest. This property enables the concrete to withstand the load from concrete cast above it, without increasing the lateral stress against the formwork (Ovarlez *et al.*, 2006). In this previous paper it is demonstrated that it is not a mater of how fast is the concrete flowing in the formwork but it is about how much time the concrete can rest before the next layer is cast. This multi-layer casting problem is associated to the flocculation rate of concrete since if it flocculates too much and the apparent yield stress increases above a critical value, the two adjacent concrete layers do not mix at all and this probably will create a weak interface in the final structure.

As a first approach of this critical time, Roussel (Roussel, 2006) proposed the following:

- the stress generated by the casting of the second layer (the new one) is of the same order as $\tau_{2nd} = \tau_0 + \eta_p \dot{\gamma}$;
- shear rate at the interface (between the two layers) is $\dot{\gamma} = \frac{V}{h}$, where V is the flowing speed of the concrete and h is the thickness of the second layer;

- since the first layer is at rest then: $\tau_{1st}(t) = \tau_0 + A_{thix} t$;
- the critical time (t_c) is the time after which the two layers will not mix and it means: $\tau_{2nd} \leq \tau_{1sd}$.

Thus,

$$\left(\tau_0 + \eta_p \frac{V}{h}\right)_{2sd} = \left(\tau_0 + A_{thix} t_c\right)_{1st} \Leftrightarrow t_c = \frac{\eta_p V}{A_{thix} h} \tag{3.20}$$

3.6 Computational model of grout flow

Recent studies are focusing on the development of computational tools to model fresh concrete flow, based on its rheological parameters. The goal is to benefit from its full potential, trying to use it in the optimization of casting technique, configuration of reinforcement and geometry of the formwork. Thus, an attempt was made in this research work to predict grout flow behaviour based on the development made in concrete.

Two types of method are available when trying to simulate the flow of a suspension such as grout. The first method consists in considering an homogeneous fluid whereas the second one takes into account the presence of the particles.

The homogeneous approach is easier to implement but is valid only when the smallest characteristic dimension (thickness of the flow, size of the mould, spacing between bars, among others) of the flow is high compared to the size of the biggest particle (Coussot *et al.* refers in (Coussot *et al.*, 1999) that it should be 5 times larger than the size of the biggest particle) and when the material stays homogeneous. According to Roussel (Roussel, 2006b) Bingham behaviour is a possible rheological model.

When the above conditions are not satisfied, it is necessary to take into account the presence of the particles (Martys, 2005), (Wallevik J., 2003). This type of approach is a lot more complex but is the only suitable technique when confined flows or flows between steel bars are studied.

Actually, it is possible to divide the previous approach into: numerical modeling of discrete particle flow and numerical techniques allowing the modeling of particles suspended in a fluid

(Roussel, 2007). However, in the following chapters, it will be used only an homogeneous approach.

Computational modeling of flow could be a potential tool for understanding the rheological behaviour of grout, mortar and concrete and a tool for mix proportioning. It can also be adopted for grout simulation of the filling and detailed flow behaviour inside a porous media as it is used in the study of concrete particle migration and formation of granular arches between reinforcement ("blocking") (Roussel, 2007).
In fact, just as numerical simulations of the loading of concrete structures allow a civil engineer to identify a minimum needed mechanical strength, numerical simulation of the injection process could allow the specification of a minimum workability of the fresh grout that could ensure the proper filling of masonry. Progresses in the correlation between mix proportioning and rheological parameters would of course result but, moreover, the entire approach to mix proportioning could be improved.

Fresh grout in this context is a suspension of particles in water. Depending on the purpose of the simulation and behaviour of the grout, the scales at which the solid components of the grout (hydraulic lime, fly ash, cement and other binders) are considered as particles or belonging to the matrix may vary.

The scale of observation on which one can reasonably observe the system and effectively consider it as a single fluid is of great importance to choose whether or not a single fluid approach is legitimate. For instance, in the concrete context (Roussel, 2007), the order of magnitude of a formwork's smallest characteristic size is around 0.1 m while the order of magnitude of the size of the coarsest particles is around 0.01 m. This means that, if, as a first approximation, the presence of the rebars is neglected, the flow in a typical formwork can be considered as the flow of a single fluid and a discrete modeling approach is thus not needed. Nevertheless, discrete methods or other alternative tools may be used by researchers and engineers for several reasons such as:

- the objective is to understand the correlation between rheology and mix composition;
- it is necessary to take into account the presence of the steel bars;
- the prediction of the segregation induced by the flow is needed.

Roussel *et al.* (Roussel *et al.*, 2007) summarizes their opinion about the efficiency of these techniques as a function of the targeted applications and according to the commercial code that they tested. Table 3.5 present that information.

Table 3.5: Applicability of some available numerical methods in the cementitious materials field (Roussel *et al.*, 2007).

Method	Simulation of testing	Simulation of casting	Blocking prediction	Segregation under flow	Thixotropy	Physical meaning	Commercial code
Single fluid simulation	Yes (except blocking)	Yes	No	No	Yes	Adequate	FLOW3D
Distinct element method	Yes	No (too many particles)	Yes	Yes	No	Poor	FIDAP
Suspension simulation	Yes	No (too many particles)	Yes	Yes	Yes	Adequate	PFC3D

Recently, Computational Fluid Dynamic codes (CFD) have also been used to model SCC or grout flow (Nguyen *et al.*, 2006), (Vasilic *et al.*, 2009). CFD is a computational technology that enables the study of the dynamics flow. Using CFD, it is possible to build a computational model that represents a system or device to be studied, based on the fluid flow physics and chemistry. The software will output a prediction of the fluid dynamics and related physical phenomena.

The governing equations of the moving fluid, like Navier–Stokes equations or Cauchy equations, are too difficult to solve analytically. Thus, approximations to those equations are necessary. Techniques like finite difference, finite volume, finite element and spectral methods are some options. CFD software deals with that.

In Chapter 8 it will be presented the numerical simulation results of the grout flow in a Marsh cone test, obtained using the CFD solving algorithm FLUENT ©, to characterize the fluidity of hydraulic lime grouts.

3.7 Concluding remarks

It is clear that there is the need to improve the techniques to optimize grouts in order to get an optimal mixture procedure, able to uniform grout and to improve its effectiveness in terms of injectability. It seems that rheology science is an important issue in that development. Regarding this literature study concerning cementitious materials, it has shown that these materials present some rheological properties that could be controlled and improved in order to become more useful in each situation. Here, the perspective is the development of grouts for masonry injection purpose.

The following table (Table 3.6) presents some rheological recommendations for concrete collected from (Roussel, 2006), (Barnes, 1997) and (Wallevik, 2009) that probably could be extrapolated for grout design.

Table 3.6: Rheological recommendations for concrete.

Situation/ requirement				**notes:**
Concrete in a tunnel	Wall:< yield stress (self-levelling)	Roof: < yield stress (self-levelling)	Floor: > yield stress (minimize segregation),< plastic viscosity (to easily place and vibrate the concrete)	t_c (critical time) between concrete layers
Casting of a silo	The concrete should flow easily, < plastic viscosity (for vibration purpose).			t_c (critical time) between concrete layers
Pumping of concrete	Yield stress should be greater than (see (1)) in order to concrete flow as plug in the pipe, otherwise segregation can occur. < plastic viscosity as possible.			$\frac{\Delta P}{L} \times \frac{D}{4} \leq \tau_0$
Under water concrete	>> plastic viscosity (to reduce flow and washout of paste).			
High strength concrete	Should present less w/b ratio. To get the same workability should present high dosage of SP. <w/b and >SP means >plastic viscosity, so it should has less yield stress than normal concrete. However, if yield stress is << then stability problems will appear and bleeding/segregation may occur especially if concrete is at rest (internal flow resistance is > if it is moving).			

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Chapter 4. The influence of the mixing procedures on the optimization of fresh grout properties

4.1 Purpose

The first part of grout research should be the definition of an optimal mixture procedure, able to uniform grout and to improve its effectiveness. This Chapter refers to the research and results from the variation of some fresh properties of natural hydraulic lime based grout that may affect injectability and stability. The study has sought to increase the understanding of the influence of the mixing procedures in grout behaviour and how it may improve some essential injection characteristics. Those results are reported in the paper (Bras *et al.*, 2009).

The correct grout selection for masonry consolidation should take into account many characteristics. This chapter analyses the variations of some fresh grout properties that may affect injectability and stability- water retention, rheological behaviour and bleeding –. For this purpose, a natural hydraulic lime grout has been considered. It is shown that there are differences in the grout behaviour when different mixing procedures are used and that fresh grout properties may be optimized if a proper mixing procedure is chosen.

Additional properties in the hardened state such as flexural, compressive strength, elastic modulus, porosity and carbonation depth were also studied and results are also presented.

4.2 Introduction

Grout injection is one of the techniques most widely used in the consolidation of masonries, to improve shear and flexural strength (Fig. 4.1). Masonry characteristics such as chemical and mineral composition, porosity, water retention, shrinkage, durability, adhesion, mechanical strengths, etc., should be taken into account in grout design. However, above all the grout should be injectable and stable for a given time.

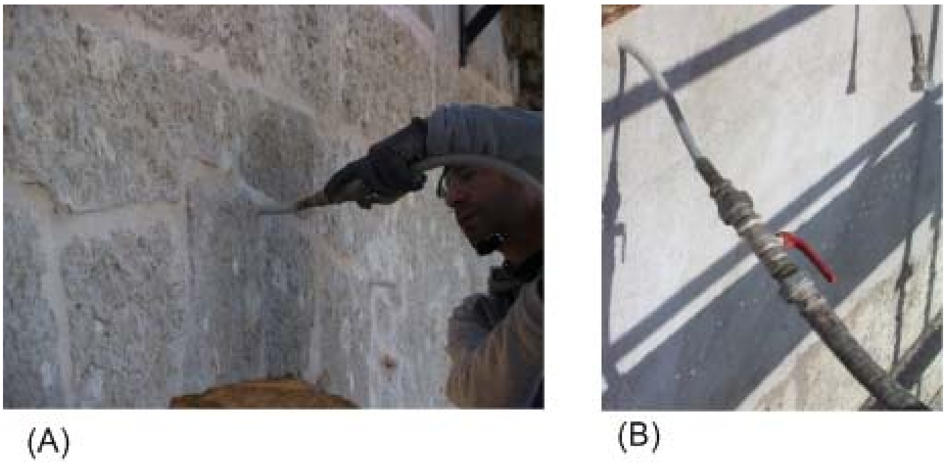

Figure 4.1: (A) and (B): Masonry grout injection.

Thus, series of laboratory tests in both fresh and hardened grouts were made in order to increase the understanding of the influence of the mixing procedure in natural hydraulic lime grout behaviour and how it may improve some essential injection characteristics.

4.3 Material Studied

4.3.1 Field of use and materials selection

The optimization of grout injectability involves an adequate control of the properties mentioned previously, along with others, which allows grouts to flow easily inside the porous media avoiding that an excessive loss of its mixing water may occur as a consequence of the medium absorption characteristics. In principle the selection of a binder to be used in grouts for injection should take into account the compatibility with the original material to be injected. Therefore, the present research program used only grouts made of natural hydraulic lime, since they present mechanical, physical and chemical characteristics closer to the original materials used in historic stone masonries – the objective of the study -, comparatively with high content cement based grouts or organic based grouts (Valluzzi, 2005).

Many parameters may affect the characteristics of hydraulic lime pastes and its injectability in particular. In order to address the influence of different mixing procedures used in the production of grouts, a mixture consisting of NHL5 natural hydraulic lime (according to EN459-1), produced in Portugal and 55% (in weight) of water has been considered here. Penetrability was not studied in the present work in spite of its relevance for injectability purposes, although it will be considered in future developments.

According to the bibliography and particularly following the recommendations proposed by (Valluzzi, 2005), a constant value of water/binder=0.55 (in weight) was used in this study. This w/b ratio represents the minimum water content necessary to grout flow in Marsh cone according to NP EN445 (see Chapter 7). The information about the various mixing procedures used to produce grouts was collected from different sources, like ASTM and EN specifications, commercial recommendations for application procedures and mixing procedures developed by other researchers. Some of these procedures were tested, along with others that were conceived during the research study. All the grouts were prepared with the same hydraulic lime composition. The main results and comments are presented in the following paragraphs.

4.3.2 Material characteristics

The hydraulic lime used is a EN459-1 NHL5 produced in Portugal by Secil-Martingança, which has the characteristics presented in Table 4.1 according with the information of the quality control system provided by the manufacturer.
Expansibility test for hydraulic lime was made according to EN 196-3.

Table 4.1: Natural hydraulic lime characteristics.

Compression resistance at 7 days (MPa)	**5,5**	
Fineness	90 μm	**12,0%**
	200μm	**1,8%**
Setting time	Start	**2h10**
	End	**7h25**
Expansibility	**0,87mm**	
Free lime	**3,70%**	
SO_3	**2,55%**	
Ignition loss	**20,07%**	

4.3.3 Mixing procedures

Before preparing the grouts, the dry hydraulic lime was hand mixed with a trowel to avoid the formation of granules. The mixing container had a capacity of 3 litres, with 0.15m diameter and a height of 0.17m. The mixer blade had a helicoidal shape, with a diameter a little smaller than the cup diameter in order to allow all the grout to be mixed (Fig.4.2).

Fig 4.2: Mixer blade used in the experimental work.

The gap at the bottom between the blade and the cup was 4mm. Ordinary tap water was used for the preparation of the grouts. The water was allowed to flow freely until a stable water temperature was reached. All mixes were prepared with a water/binder ratio of 0.55. The measured density for all mixes was 1700 ±5 kg/m^3. The following mixing procedures were tested:

(MIX1): 2/3 of binder is added to the water and mixed at 2100 rpm during 2 minutes. The mixer is stopped and the remaining binder is added. After all materials have been added the mixing continues for 3 min. This mix was conceived by the authors during the study.

(MIX2): After placing all the water in the container the whole binder is added, followed by a 4 min mix at 2100 rpm. This mixing procedure was based on the ASTM procedure (ASTM C938-02), but instead of mixing only during 3 minutes more time was used to reduce the variability of rheological properties (Eriksson *et al.*, 2004).

(MIX3): The whole binder is added to 3/4 of the water and mixed during 2 min at 2100 rpm. The remaining water is added within 30 seconds without stopping the mixer. After all the materials have been added the mixing continued for an additional 4 min (Toumbakari, 2002).

(MIX4): The whole binder is added to 2/3 of the water and mixed at 2100 rpm during 2 minutes. The remaining water is added within 30 seconds without stopping the mixer. After all materials have been added the mixing continued 3 min. This procedure was adapted from a technical specification for grouts conceived by Sika.

(MIX5): After placing all the binder in the container and mixing during 60 seconds at a very low speed (not determined) the water is progressively introduced during 2 minutes without stopping the mixer. After that, the mixer is stopped and the grout is mixed with a trowel for 30 seconds in order to scrap the container. The grout is then mixed during 90 seconds at 1800 rpm (Rosquoët *et al.*, 2003).

After preparation all the mixes were sieved with nº16 ASTM (1.18mm) sieve to discard the larger grains. The previous mixture information is synthesized in Table 4.2.

Table 4.2: Different mixing procedures tested for the same grout composition (NHL5 with w/b=55%).

Mixture Procedure	binder addition	water addition	mixer speed	mixing time	notes:
MIX 1	66.7%	100%	2100 rpm	2'	Water is placed into the container first
	33.3%	-	0 rpm	30''	
	100%	100%	2100 rpm	3'	
MIX 2	100%	100%	2100 rpm	4'	Water is placed into the container first
MIX 3	100%	75%	2100 rpm	2'	Water is placed into the container first
	-	25%	2100 rpm	30''	
	100%	100%	2100 rpm	4'	
MIX 4	100%	66.7%	2100 rpm	2'	Water is placed into the container first
	-	33.3%	2100 rpm	30''	
	100%	100%	2100 rpm	3'	
MIX 5	100%	-	low speed	1'	Binder is placed into the container first
	-	100%	low speed	2'	
	mix with a trowel		0 rpm	30''	
	100%	100%	1800 rpm	90''	

The determination of mixer speed was made using a stroboscope, which is an instrument used to make a cyclically moving object appear to be slow-moving, or stationary. A stroboscope uses a flash lamp driven by an electronic oscillator. The flash lamp is a xenon bulb. The oscillator triggers the lamp at a steady rate, settable from a few times per second to thousands of times per second. The flash lamp has a reflector to increase its brightness and to make the flash more directional. The principle is used for the study of rotating, reciprocating, oscillating or vibrating objects. Thus, it was adopted in rotation speed measurement of mixer in each studied case.

Figure 4.3: Stroboscope used in the experimental work.

4.4. Water retentivity

4.4.1 Procedure

It is not always safe to pre-wet the masonry by injecting water before the grout injection, in order to make the grout flow inside the porous medium easier minimizing water loss. The mechanical strength of the pre-wetted samples is very poor, so, the grout must have the capacity of retaining the mixing water for a long time in order to preserve its properties and to make possible the flow. Water absorption out of the grout into the masonry may be a possible injection blocking mechanism. When meeting a dry and absorptive masonry it is important to use a grout with excellent water retaining properties. Thus, a stable grout will be more able to retain the water.

To determine the water retentivity – the ability of a grout mixture to retain its mixing water - all mixtures were tested according to a procedure based on ASTM C941-02 (ASTM C941-02). A pressure of 94±0.5 kPa is applied to the system where the grout is introduced, for the duration of the test. The grout is placed inside a filtering funnel until it reaches the top. A stopcock is placed between the funnel and the graduated cylinder, which is opened at the same time the stop-watch is started. The time needed to remove 30 ml of water from the grout sample is registered. All grouts were tested within 1 min after being mixed (Fig.4.4).

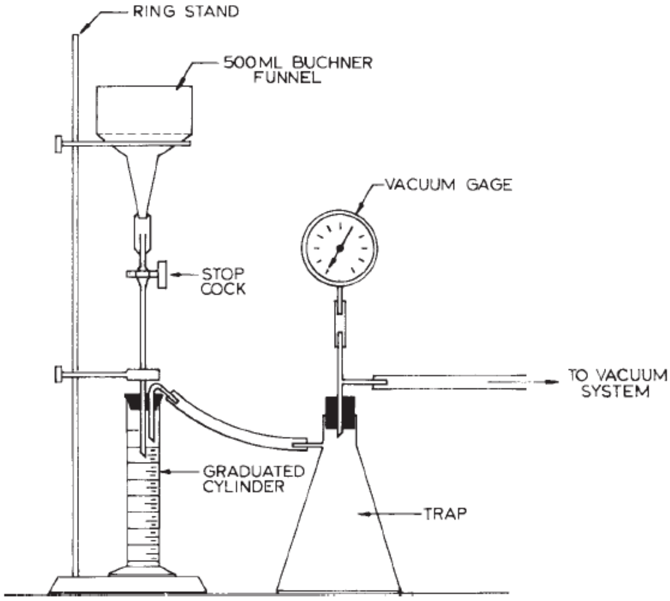

Figure 4.4: Device adopted for measurement of the water retentivity (ASTM C941-02).

4.4.2. Results and comments

Grout characterization using only deterministic parameters is complicated because the material presents too many changes of properties. Some research works developed until now presented grouts properties based only in single tests, while others are based on the repetition of some tests of each grout mix. The latter referred the existence of substantial variations, for example coefficients of variation (CV) of 2-22% are mentioned by (Svermova *et al.*, 2003). This uncertainty experienced on grout properties makes it difficult to foresee the real characteristics of the grout. Those variations may explain some contradictory results obtained when comparing results of single tests (Eriksson *et al.*, 2004).

To overcome this difficulty the present research was based on the use of at least five samples of each grout mixture type, allowing coefficients of variation that are within a reasonable range. The results obtained for the values of water retention time of the mixtures are presented in Table 4.3.

In spite of the scattering of results, which lead to the need of conducting several repetitions to minimize the uncertainty, the table shows substantial differences between the different mixing procedures.

According to the water retentivity results, the worst mixtures are MIX5 and MIX2 and the better ones are MIX3 and MIX4. As it can be seen, MIX2 and MIX5 have similar procedures and results, since all the water is placed with the whole binder in just one step. However in MIX2 the binder addition to water is quicker, while in MIX5 the water is added slowly to the binder.

MIX5 was prepared following a complex procedure since it has several steps and it is difficult to mix the water with the binder without producing a paste with sediments at the bottom. Besides that, the slow mix velocity and the slow water addition enhance grout sedimentation and heterogeneity. Thus, it was concluded that it is better to add the binder to the water previously placed in the container (and not the opposite).

On the contrary of what was expected, the two steps of MIX1 procedure (2/3 of binder added to the whole water and mixed, plus 1/3 of binder added to the previous paste and mixed again) were not the best way to improve the water retentivity, in spite of its values being higher than those of MIX2.

Comparing MIX3 and MIX4 there is a progressive improvement of water retention from MIX4 to MIX3, which also means that a progressive addition of water improves the grout performance together with a greater mixing time.

The addition of 3/4 of water in the first mixture phase of MIX3 leads to a good improvement of retention capacity comparatively with MIX4 (addition at the first phase of 2/3 of the whole water used to produce grout). This evidence suggests the existence of an optimal dosage of water (somewhere in between of MIX3 and MIX4) that should be added in the first mixture phase if a maximum water retention capacity is intended.

Thus, the authors decided to test a new mixture procedure, MIX6, similar to the previous MIX3 and MIX4 but using the arithmetical average of the first water quantities used in the previous mixtures procedures ((3/4+2/3)/2=17/24), which can be defined as follows:

(MIX6): The whole binder is added to 17/24 water and mixed during 2 minutes. The remaining water is added within 30 seconds without stopping the mixer. After all materials have been added, mix for 4 min at 2100 rpm.

Table 4.3: Water retention time results for different mixing procedure.

	Water retention time					
	MIX1	**MIX2**	**MIX3**	**MIX4**	**MIX5**	**MIX6**
	1029s 1026s 1031s 1115s 876s 862s 880s	910s 930s 915s 769s 799s 750s	1747s 1830s 1680s 1170s 1658s	1154s 1302s 1292s 1380s 1070s 1265s	787s 739s 877s 846s 931s	1588s 1841s 1680s 2190s 1770s
Average:	**974.1s** **16.2min**	**845.5s** **14.1min**	**1617.0s** **27.0min**	**1243.8s** **20.7min**	**836.0s** **13.9min**	**1813.8s** **30.2min**
CV:	10%	10%	16%	9%	9%	13%

The analysis of the results shows that there is an improvement in the water retention of natural hydraulic lime grouts if the first amount of water to be mixed with the binder is optimized, as can be seen from the results obtained with MIX6.

As previously noted a stepped addition of binder or water may improve the water retention of natural hydraulic lime grouts. However, the results point out to the need of adding the binder to the water and not the opposite to avoid the formation of granules. The addition of water to the whole binder in MIX6 promoted a better contact between the particles of lime and water molecules, which improved the microstructural connexion, the adhesion between particles and the connexions of porous network in the fresh grout. Hence a better water retention was achieved. This higher water retention creates a better flow inside the porous medium where the grout is injected.

The optimization of this property diminishes the negative effect that flow in porous media produces in the rheological behaviour of a grout. A highly absorbent medium is able to drastically reduce the w/b of a grout when the flow occurs. If the w/b ratio is diminished the viscosity increases and the flow may be interrupted inside porous media, leading to an ineffective injection procedure and lack of compactness improvement.

4.5. Study of the Rheological behaviour

4.5.1 Procedure

To characterise the rheological behaviour of freshly mixed hydraulic lime grouts, all the mixtures were tested using a Brookfield viscometer LV DV-II+PRO with an attached guardleg (Fig.3.20) and disc spindles placed at the centre of the sample container used during the test. The same cup was used in all tests (Rc= 47.5 mm) filled with 500 ml of fresh grout. The spindle has a radius of 6.3 mm and was rotated at specific speeds, selected by the program used.

Two different programs were selected to test the grouts. Program 1 enables to understand what happens to a grout when subjected to different shear rates. Program 2 gives information about possible grout coagulation and its behaviour at a constant shear rate.

Program 1 consisted in inputting different angular velocities (rpm) to the fresh grout 10 minutes after mix preparation, from 0 rpm to 200 rpm with steps of 5 rpm each 15 seconds during 10 minutes. The output data were the apparent viscosity, recorded every 15 seconds.

Program 2 consisted in inputting a constant angular velocity of 50 rpm (constant shear rate) to the previous mix during 2h after program 1 ended. The output data were the apparent viscosity, recorded every 10 minutes.

At least three tests were conducted for every mixing procedure. Only average values for each mix are presented. The RH of the laboratory was 50% and the temperature 23±2 ºC.

4.5.2 Results and comments

The recorded average values of viscosity *versus* angular velocity for all mixtures using Program 1 are presented in Fig. 4.5.

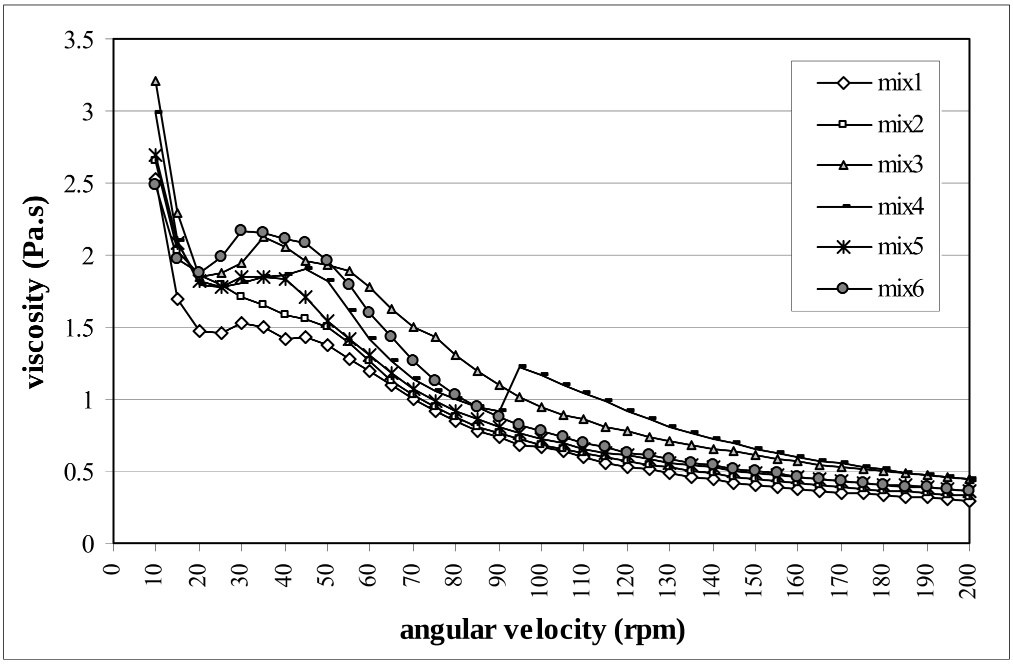

Figure 4.5: Values of viscosity *versus* angular velocity for all mixtures using Program 1.

There is a decrease of grout viscosity for all the mixes when angular velocity increases as can be seen in Fig.4.5. This behaviour of the hydraulic lime grouts can be compared to a shear-thinning fluid (viscosity decreases as shear rate increases), as it occurs in cement grouts with low w/b ratios similar to those used in the present work (Rosquoët *et al.*, 2003).

Between 20 and 50 rpm all mixtures present a viscosity increase. A possible explanation for that may be a chemical hydration reaction that is enhanced at this rpm range. This viscosity increase could also be explained as a transition phase from the initial random distribution of particles to a preferential orientation that is achieved at higher angular velocities. A shear-thickening behaviour also occurs in this phase.

Within this range of rpm MIX6 is the one that has the greatest viscosity values followed by MIX3 and MIX4. Analysing the performance of those three mixtures procedures in water retention tests, it could be said that different levels of free water inside fresh grout are obtained. In this mixes (MIX6, MIX3 and MIX4), the free water is more limited in comparison to the remaining mixture procedures and flow is more difficult. Probably for that reason between 20 and 50 rpm those grouts present a higher viscosity.

At the end of the test (>180 rpm), all grouts present a constant viscosity. This is identified as the second Newtonian region (≈0.35 Pa.s). The reason why mixtures present a shear-thinning behaviour may be explained by the shape of grains (Toumbakari, 2002). Spherical particles (as it is the case of some types of cement) may lead to a grout that exhibits almost a Newtonian behaviour, while particles with elongated shape (as is the case of the natural hydraulic lime grouts studied here) may lead to a grout with shear-thinning behaviour (Fig.4.6 and Fig.4.7).

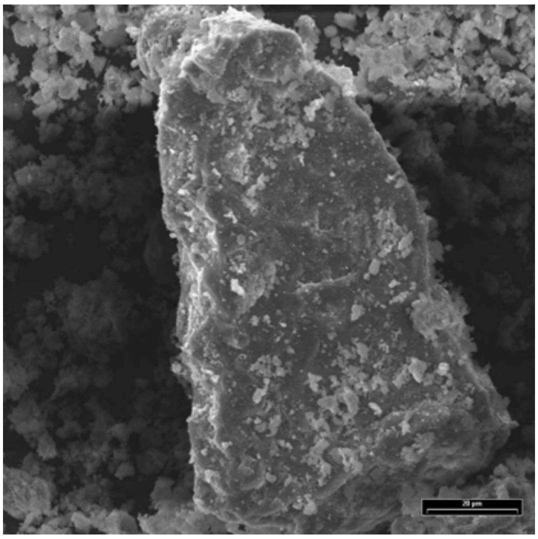

Figure 4.6: SEM image of natural hydraulic lime particle at 750x.

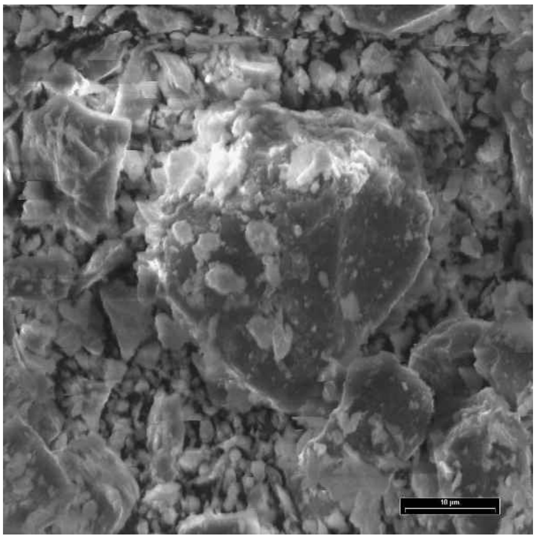

Figure 4.7: SEM image of cement type I particle at 1500x.

With the increasing shear rate the elongated particles tend to a preferential orientation inside the fresh grout when flow occurs. In this way there is a viscosity decrease, which explains the previous results.

The smallest viscosity observed for a natural hydraulic lime grout with 55% water/binder ratio (≈0.35 Pa.s) is at least 1 order of magnitude greater than the plastic viscosity detected when using higher w/b ratios (w /b=0.80), as referred in **(**Eriksson e*t al.*, 2004**)** .

Program 1 begins 10 min after the preparation of the mixtures and runs for 10 min, after which program 2 is run. This means that program 2 started 20 minutes after the mix preparation. Since it only records data 10 min after the beginning of the program, this means that the first registered values were obtained only 30 minutes after the mixes were prepared. The recorded average values for all mixtures using Program 2 are plotted in Fig. 4.8.

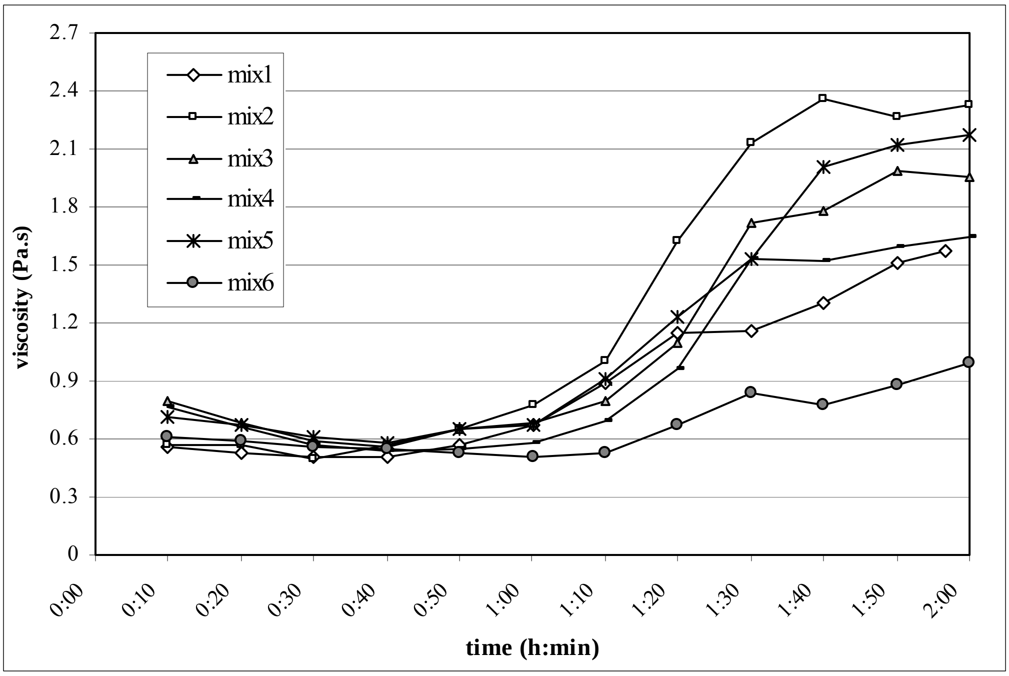

Figure 4.8: Values of viscosity *versus* time for all mixtures using Program 2.

All mixes presented a low and relatively constant viscosity for a constant shear rate until 1h30 (1h10+20’) after grout preparation. However, after that time shear-thickening behaviour occurs for all of them with a substantial viscosity increase with time. This increase is not the same for all the mixing procedures. MIX 2 and MIX 5 present the bigger slope and once again MIX 3, 4 and 6 exhibit the smallest viscosity. The increase of viscosity of mixes 2 and 5 with time is attributable to continuing coagulation maybe due to instabilities. On the other hand, the good wetting of mixes 3, 4 and 6 retard coagulation and help in limiting the viscosity increase. Another possible explanation of the viscosity increase may be the beginning of the chemical reactions that lead to the hardening of the grouts. With the beginning of this reaction the microstructure becomes denser and the skeleton more resistant. As result, the fluid shows bigger opposition to a given shear rate.

MIX6 is the one that presents the smallest viscosity increment along the time which means that its stability is greater and its injectability should be better for a larger amount of time. MIX4 and MIX3 present the second best results after MIX6, what is in agreement with the water retention results. MIX2 and MIX5 are the worst in this point of view because they present the greatest viscosity at the end of the test.

Again it is confirmed that a variation in mixing procedures leads to different rheological behaviour of grouts based on natural hydraulic lime.

4.6. Study of grout bleeding versus stability

4.6.1 Procedure for bleeding test in freshly mixed grouts

The bleeding test used in this research was based in ASTM C 940. According to this standard 800 ml of freshly mixed grout was poured into a 1000 ml glass graduated cylinder and covered. The height of bleed water was noted after complete sedimentation. The final bleeding was calculated according to the expression (ASTM C940-98a):

$$\text{Final bleeding } (\%) = \frac{V_w}{V_1} \times 100 \qquad (4.1)$$

where,

V_W = volume of decanted bleed water, ml

V_1 = volume of sample at beginning of test, ml

The main purpose of the test was to evaluate the influence of mixing procedures on the stability of the grouts. An excess of bleeding in a grout means that there is a substantial amount of free water on the surface of the unset grout. A drawback of this test is that it leads to discrete results that do not allow checking the evolution of density gradient of the grouts.

Consequently, a new test procedure - the stability test- was developed, based on the test proposed by Van Rickstal (Van Rickstal, 2000) aimed at checking the density variation of a grout in resting conditions (which are unfavourable as far as stability is concerned). For this test 500 ml of freshly mixed grout was poured into a 500 ml glass graduated cylinder placed under a balance. A cylindrical object with a volume of 65 cm^3 and a mass of 133,210 g suspended from the balance was placed inside the cup with fresh grout (Fig. 4.9). The mass variation was recorded with time.

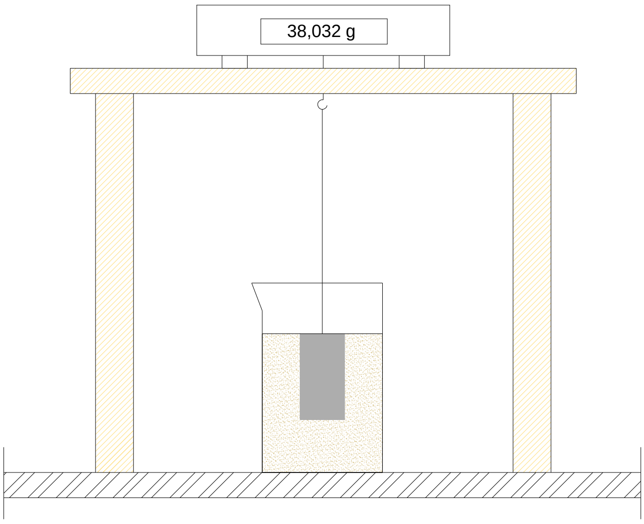

Figure 4.9: Configuration adopted for the stability test.

Three samples for each mixture were tested at a temperature of 23±1ºC and 40% RH. Based on the principle of Archimedes the difference between the weight of the object and the buoyancy force was recorded.

4.6.2 Results and comments

The results obtained for the evaluation of bleeding of the six grout mixes, based in ASTM C 940, are presented in Table 4.4, where it can be seen that the differences of bleeding among mixes are not relevant and virtually no bleeding is detected in the tested specimens.

The results of the stability tests are plotted for each mix in Fig.4.10 as percentage of initial density versus time.

Table 4.4: Final bleeding for all mixtures procedure tested.

Mix designation	Final bleeding (%)
1	0.33
2	0.66
3	1
4	0.66
5	None
6	None

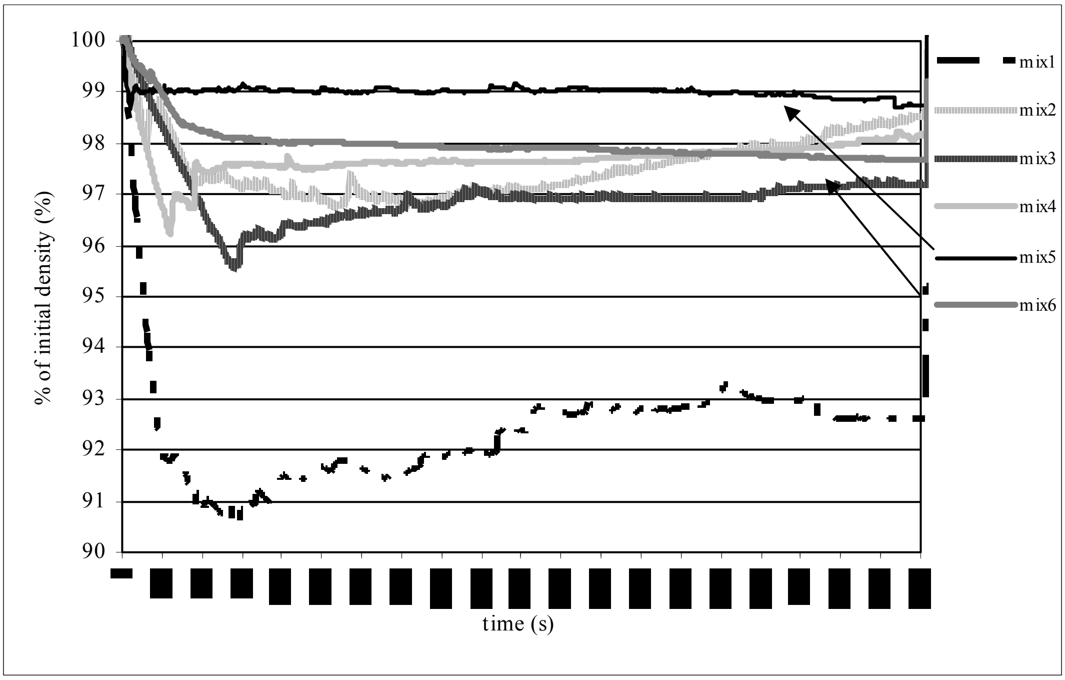

Figure 4.10: Percentage of initial density *versus* time for the 6 mixtures procedure tested, according to the proposed stability test.

In general terms the density of a fluid is determined as an average for a given volume. This does not mean that within the several layers of the fluid the density remains constant. In fact, the density of the analysed layers differs as a consequence of the different levels of stability of the grouts. If density variations are small the grout is stable and the bleeding effect is minimum.

The results obtained show differences between the studied mixes, MIX6 and MIX5 being the most stable during the period of 1.5 h used for the tests. The worst results were obtained by MIX1 which presented an ample gradient of variation. Unexpected results were obtained for MIX5, for which a similar behaviour could be expected.

This behaviour is probably due to different connections of the porous networks of the fresh grouts when mixed in different ways. According to (Shannag, 2002) the bleeding effect leads to a lack of durability, weakness and porosity, because of the formation of uncontrolled open channels within the grout mass.

4.7 Study of grout hardened properties

4.7.1 Procedure for grout strength test, porosity and carbonation analysis

Together with the previous tests, grout strength determination was made to the different mixing procedures tested (MIX 1, 2, 3, 4, 5 and 6). In order to determine mechanical characteristics of the formulated grouts a testing campaign was undertaken and all samples were submitted to flexural and compressive strength tests following standard (NP EN 1015-11). The dynamic modulus of elasticity was determined following (Fe 08 DEC/UNL, 1996). Testing was performed at the ages of 28 days and the load was applied without shock at a uniform rate of 30N/s for flexural strength. For compressive strength determination, the load was applied also without shock and was increased continuously at a rate of 300N/s. For both situations, the failure occurred within a period of 30s to 90s.

In order to analyse grout porosity when different mixing procedures are used (Fe 02 DEC/UNL, 1996) was adopted. The carbonation depth was determined using a phenolphthalein pH-indicator (Fe 28 DEC/UNL, 1996).

4.7.2 Results and comments

The average measurements of flexural, compressive and dynamic modulus of elasticity are presented in Table 4.5. It shows that it seems there are some differences in strength and elastic modulus results for each mixture tested. However, some results dispersion may be found for the six grout samples tested in each mixing procedure. So, no explanation to this phenomenon is obtained unless the fact that probably some of the mixing procedures lead to different air retention content distribution inside grout. Since no air content was measured in fresh grout, porosity in the hardened state was measured together with carbonation depth in an attempt to get some information about it (Table 4.6).

Table 4.5: Strength and elastic modulus results for different mixing procedure for NHL5 based grout with w/b=0.55.

NHL5 w/b=0.55	Strength at 28 days				E_{28d} (MPa)	CV (%)
	Flexural (MPa)	CV (%)	Compressive (MPa)	CV (%)		
MIX1	1.8	9.0	5.4	6.0	3208	23.0
MIX2	1.1	23.0	6.9	23.0	2824	30.0
MIX3	0.6	18.0	6.4	7.0	1186	34.0
MIX4	0.1	30.0	6.1	15.0	1196	24.0
MIX5	1.7	25.0	11.1	5.0	2732	21.0
MIX6	1.0	20.0	6.1	11.0	2900	29.0

Table 4.6: Porosity and carbonation depth at 28 day for NHL5 based grout with w/b=0.55.

NHL5 w/b=0.55	Porosity (%)	CV (%)	Carbonation depth (mm)	CV (%)
MIX1	52.0	1.9	11.0 mm	5.6
MIX2	52.6	0.3	9.3 mm	4.0
MIX3	52.0	0.8	9.6 mm	1.5
MIX4	51.6	1.0	7.2 mm	13.0
MIX5	51.4	3.0	5.6 mm	2.6
MIX6	52.0	2.9	5.5 mm	3.1

The analysis of porosity values leads to the conclusion that it does not change if the mixing procedure is modified. However, carbonation depth became higher in MIX1 and smaller in MIX5 and 6. Carbonation starts from the grout surface and penetrates inside grout microstructure faster if there is a porous network that contacts the surface.

According to these previous assumptions, it seems that probably MIX5 and 6 leads to a porous network less extensive in deepness than MIX1, 2 and 3. Coefficients of variation show that there is a certain precision in those measurements to get a conclusion.

4.8 Discussion and conclusions

Several mixing procedures were tested in order to understand their influence in natural hydraulic lime grout behaviour. Fresh grout properties like water retention, rheological behaviour and stability were observed. Hardened properties such as flexural, compressive strength, elastic modulus, porosity and carbonation depth were also studied. The following points have to be emphasized.

The resistance of the grout to forced bleeding was determined according to the procedure described above using a pressure below atmosphere conditions simulating the pressure decrease of an injected porous substrate, where suction is present probably due to the surface tension. The adopted procedure shows that there is different grout behaviour when different mixing procedures are adopted. According to the results, there is an improvement in the water retention of natural hydraulic lime grouts if the first amount of water to be mixed with the binder is optimized, as can be seen from the results obtained with MIX6. Water retention is improved by using a stepped addition of water. This higher water retention creates a better flow inside the porous medium where the grout is injected.

The results obtained with MIX3 indicate that this mixing procedure (where the first parcel contains 3/4 of the all water) leads to water retention higher than MIX4 but lower than MIX6. The results of MIX4 indicate that this mixing procedure (where the first parcel contains 2/3 of the all water) leads to water retention lower than MIX3 and MIX6 but higher than remaining mixing procedures.

It can be also observed that the period of mixing is important: a greater mixing time leads to a better wetting, which partially explain the good behaviour of MIX3 comparing to MIX4.

Since water retention is a function of the absorption characteristics of the particles inside the grout, their shape and relative location are crucial for the explanation of the phenomena. Given the fact that in the present case the particles are the same, their relative position should be responsible for the differences obtained. Thus, it seems that the quality of dispersion determined the water retention capacity since it allows more particles to be wetted and therefore more water is adsorbed on the particles surface and less water is free to bleed. Less space between binders particles – as it is the case of those with less water added in the first parcel - should enable a better water adsorption, as it is the case of MIX4. But it should also be taken into account that a suitable distance between binder particles allows not only a better water retention but also their easier orientation following preferential patterns. Since natural hydraulic lime particles are elongated, if they are placed parallelly their water retention due to the adsorption forces between its surfaces will be higher than in the other cases. This could explain the better results obtained with MIX6.

The procedure adopted to study the rheological behaviour of fresh natural hydraulic lime grouts shown that MIX6 is the one that presents the smallest viscosity increment along time (for a constant shear rate), which means that its stability is greater (less mixture coagulation) and its injectability should be better for a larger period of time, comparatively to the other mixing procedures tested. Consequently, the hardening of MIX6 is slower, enabling two different layers of injected grout to combine easily during the injection phase. The microstructure of the first grout layer becomes denser and the skeleton more resistant. However, this is a slow process and the penetration of the second layer becomes less complicated. A wider range of injection time is reached with MIX6 when compared with the other mixing procedures tested. Besides that, a substantial improvement of the medium homogeneity may be obtained.

The bleeding test presented no relevant differences between the mixing procedures for the w/b=0.55 adopted. However, if the stability test is done differences arise, showing that MIX6 and MIX5 are the most stable. According to Fig. 4.5 there is more free water in MIX1 which leads to less viscosity but also to a least stable grout, as confirmed by Fig.4.10. This fact suggests that the addiction of water to binder (and not the opposite) produces an inefficient mixture.

The previous points show that different mixing procedures present an important effect on properties of fresh grouts. Those properties are crucial and should be controlled in injection grouts.

NHL5 based grout made using MIX6 (with w/b=0.55) present a flexural strength equal to 1.0MPa and a compressive strength equal to 6.1MPa, tested at 28days. Elastic modulus is about 2900MPa.

The analysis of porosity and carbonation depth of different grout tested leads to the conclusion that probably some of the mixing procedures present different air retention content distribution inside grout. Porosity is constant in each mixture together with grout composition. Thus, probably carbonation evaluation becomes an effective test since this phenomenon starts from the grout surface and penetrates inside grout microstructure faster if there is a porous network that contacts the surface. According to these previous assumptions, it seems that probably MIX5 and 6 lead to a porous network less extensive in deepness than MIX1, 2 and 3.

A detailed study concerning air content in fresh grout should be conducted in the future.

This study (Bras *et al*., 2009) analyses the variations of some fresh grout properties - water retention, rheological behaviour and stability - observed in a series of laboratory tests as a consequence of different mixing procedures. It has sought to increase the understanding of the influence of the mixing procedure in natural hydraulic lime grout behaviour and how it may improve some essential injection characteristics. Some hardened properties were also tested to check material strength and main differences between mixing procedures.

The general results show that there are differences in the grout behaviour when different mixing procedures are used and that fresh grout properties may be optimized if a proper mixing procedure is chosen. According to this experimental test an optimal mixture procedure (MIX6), for natural hydraulic lime grout, should be the following one: the whole binder is added to 17/24 water and mixed during 2 minutes. The remaining water is added within 30 seconds without stopping the mixer. After all materials have been added, mix for 4 min at 2100 rpm.

4.9 References

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Chapter 5. Effect of environmental temperature and fly ash addition in hydraulic lime grout behaviour

5.1 Purpose

The definition of an optimal mixture procedure is crucial for a best grout performance. The period of mixing is important since a larger mixing time leads to a better wetting. Stability tests show that there are different results when using different grout mixing procedures. Some of them lead to more free water than others and this means that a less stable grout will be obtained, which is not good (Bras *et al.,* 2009).

Besides that, other parameters, such as grout rheological behaviour and water retention capacity, change with the selected procedure. All these facts suggest that the addiction of water to binder (and not the opposite) produces an inefficient mixture. Thus, for all the following NHL5 based grout the same procedure (MIX6) was adopted.

The following research work was planned to get an insight into rheological properties that may have an impact in grout behaviour. Time independent and dependent fresh grout properties that may change due to the influence of environmental temperature and due to fly ash addition in fresh NHL5 grouts were evaluated. However, in this chapter it will be presented only the results and conclusions concerning time independent properties. According to grout masonry injection requirements, an attempt was made to achieve an optimal grout composition by using the Taguchi method.

The results presented in (Bras *et al.*, 2010) show that fly ash is capable of substantially changing the grout rheological parameters, while the effect of temperature is negligible for the tested time independent properties (such as yield stress, plastic viscosity, consistency and power-law index).

Mechanical strength and elastic modulus of the tested grouts are also presented together with porosity analysis and shrinkage performed on the best grout composition obtained.

5.2 Introduction

Fly ash is a by-product of the combustion of pulverized coal in thermal power generation. It has been used in grouts since it reduces the costs and the environmental impacts (Ozcan Tan *et al*., 2005). Besides that, it is expected that the replacement of a certain amount of the hydraulic binder by fly ash will improve grout durability, which could contribute to a good performance of old masonries (Binda *et al.*, 2003), (Penazzi *et al*., 2001).

In the presence of water, pozzolanic materials like, fly ash, react with calcium hydroxide, a basic component of hydrated natural limes, to form silicates and aluminates. Probably the calcium hydroxide consumption by fly ash minimizes the risk of ettringite and thaumasite formation (Collepardi, 1999).

The spherical shape and the small size of fly ash, when compared to natural hydraulic lime, increase grout density, as confirmed by (Sonebi, 2006) and minimizes material voids, therefore contributing also to a greater durability.

The injection of appropriate grouts represents a precious way to repair and strengthen stone masonry provided the original and new materials are accurately controlled and selected. Binda *et al* have developed and implemented a methodology based on three principal steps (Binda *et al*., 1997), (Binda *et al*., 2002):

1) Laboratory characterization of the materials sampled from the masonry and choice of grouts suitable for injection through an injectability test;
2) Injection on site of check points;
3) Control of the injection efficacy by flat jack test and survey of the penetration and diffusion of the grout.

Failure of consolidation technique may occur in the following situations: (i) if the injected parts present heterogeneous strength and stiffness; (ii) there is poor penetration and diffusion of grout due to a difficult injectability or injection technique; (iii) due to segregation and grout shrinkage; (iv) if there is grout physical and chemical incompatibility with masonry original materials (Binda *et al.*, 1997). Therefore, consolidation quality depends upon the characteristics of masonry materials and on grout behaviour. Fresh grout properties seem to be as important as the ones in the hardened state since grout consistency is an essential characteristic to allow the filling of voids. Thus, the optimization of grout injection is a task of major importance.

Grout flow properties are affected by a large number of parameters, including not only binder content, type and composition, type and dosage of superplasticizer and water/binder content (w/b), but also environmental conditions (mainly moisture and temperature), water temperature, mixing time, mixer type, energy and sequence of mixing (Eriksson *et al.*, 2004), (Fernàndez-Altable *et al.*, 2006), (Bras *et al.*, 2009). Such a large variety of factors plays a significant role on grout rheology.

A suitable grout composition, together with the flow capacity and the ability to fill the voids, will regulate the consolidation quality. From this point of view, the evaluation of this capacity involves the control of a few important values, such as yield stress and plastic viscosity, among others. The type and number of parameters necessary to characterize a fluid like grouts depend on their type of rheological behaviour.

Barnes and colleagues (Barnes *et al.*, 2001) state that for shear-thinning non-Newtonian fluids the general shape of the curve representing the variation of viscosity with shear rate is composed by two baselines (constant viscosity values) for low and high shear rates. The curves indicate that in the limit of very low shear rates (or stresses) the viscosity is constant, whilst in the limit of high shear rates (or stresses) the viscosity is again constant, but at a lower level. These two extremes are sometimes known as the lower (first Newtonian region- η_0 (Pa.s)) and the upper Newtonian regions (second Newtonian region- η_∞ (Pa.s)).

Since this research analyses the variations of some fresh grout properties of natural hydraulic lime grouts with finer powders like fly ash and knowing that the behaviour of hydraulic lime grouts can be compared to a shear-thinning fluid (Bras *et al.*, 2009), as it occurs in cement grouts with low w/b ratios similar to those used in the present work (Rosquoët *et al.*, 2003), the rheological model adopted was the Sisko model (Eqn. 5.1) (Barnes *et al.*, 2001):

$$\eta = \eta_\infty + K_2 \dot{\gamma}^{n-1} \tag{5.1}$$

where K_2 is called the consistency (Pa.s^n) and *n* is called the power-law index.

An increasing of K_2 brings to a grout viscosity increase. On the other hand, if a grout has a shear-thinning behaviour *n* must be less than 1. For this mathematical domain, a convergence to *n*=1 leads to a less shear-thinning and more Newtonian behaviour. If *n* is set equal to zero, the previous equation change to

$$\eta = \eta_\infty + \frac{K_2}{\dot{\gamma}} \tag{5.2}$$

which can be modified to

$$\tau = \tau_0 + \eta_p \times \dot{\gamma} \tag{5.3}$$

called the Bingham equation, where τ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate, τ_0 is the yield stress (Pa) and η_p is the plastic viscosity (Pa.s). Since the Bingham model is only valid for *n*<0.20 and $\frac{\eta_0}{\eta_\infty} > 10^9$, the modified Bingham equation was adopted

$$\tau = \tau_0 + \eta_p \times \dot{\gamma} + c \times \dot{\gamma}^2 \tag{5.4}$$

where *c* is an insignificant constant.

Coussot *et al* in (Coussot *et al.*, 2002) refers that yield stress fluids can be defined as fluids that can support their own weight and it means that the fluid on an inclined plane will not flow if the slope is below a critical value (Fig. 5.1).

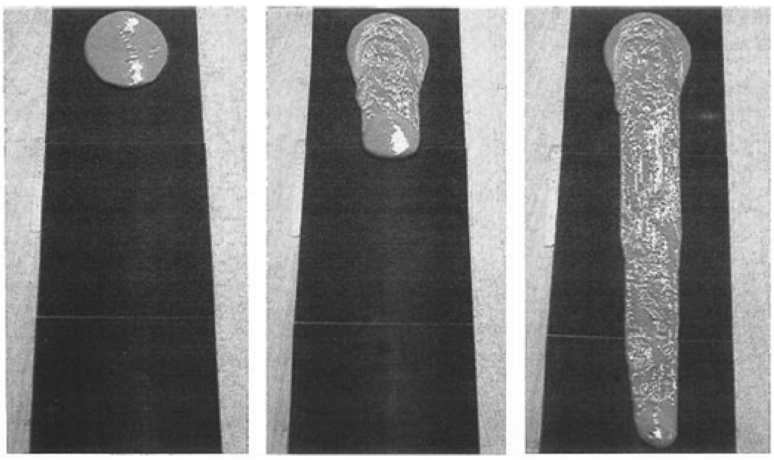

Figure 5.1: Avalanche flow of a clay suspension over an inclined plane covered with sandpaper. The suspension was pre-sheared and poured onto the plane, after which it was left at rest for 1 h. The pictures are taken at the critical angle for which the suspension just starts to flow visibly (Coussot *et al.*, 2002).

According to Roussel (Roussel, 2007), from a practical point of view yield stress is probably associated to the ability of the grout in filling up the voids and its ability to flow when a given shear stress is applied. The knowledge of the yield stress enables the understanding 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. Thus, yield stress may be used as a control parameter for the application of injection grouts.

Plastic viscosity may be associated to the velocity at which a given grout will flow after the beginning of flow. Like yield stress, plastic viscosity may also be used as a prediction factor of whether or not a grout will be able to be pumped. Less viscosity leads to a higher flow velocity inside the porous medium.

The present study concerns natural hydraulic lime grouts with fly ash and some relevant rheological properties are analysed. The main goal is to develop an optimal grout composition for injection purpose, taking also into consideration the effect of environmental temperature in those fresh grout properties.

From a rheological point of view, a reduction of the yield stress value was detected when cement was partially replaced by pulverised fly ash in cement based grouts (Sonebi, 2006). Fly ash improves the contact between the particles of cement by ball bearing effect and reduces the friction forces, therefore reducing the applied shear stress needed to initiate flow. Mirza *et al* (Mirza *et al.*, 2002) noticed the improvement of stability and the reduction of drying shrinkage in cement based grouts with fly ash, which is a positive trend. A limited shrinkage of a grout is desirable since it reduces the probability of micro-cracking along the interfaces between grout and the original materials of the wall.

Taking into consideration all these points, several fly ash/binder ratios were tested in order to understand their influence in natural hydraulic lime composites grout behaviour.

In situ grout application may occur at several environmental conditions, which may lead to different grout injection capacities, as reported by Eriksson *et al.* (Eriksson *et al.*, 2004). These authors noted the influence of water temperature as a factor able to influence grout properties. Considering that the application of a grout may begin early in the morning and continue throughout the day during several days, the variations of temperature and relative humidity become self evident. Other authors have analysed the same influence, as it is the case of Fernàndez-Artable *et al.* (Fernàndez-Altable *et al.*, 2006) that identified a relation between the grout rheological behaviour and the environmental temperature, stating that one of the key issues in understanding the interactions between cement particles (or their hydrates) and surfactant agents such as superplasticizers is the influence of environmental temperature on the early stage evolution of cement pastes.

Analyses of some relevant rheological properties for natural hydraulic lime grouts, such as yield stress, plastic viscosity, consistency and power-law index were made. The study envisage to increase the understanding of the influence of environmental temperature in those fresh grout behaviour and the influence of finer powders, like fly ash, in grouts for injection purpose. Mechanical strength, elastic modulus and porosity are also studied in order to check the influence of fly ash in those hardened properties.

5.3 Experimental

5.3.1 Material studied

5.3.1.1 Scope

Compatibility with the original material to be injected should be taken into account when choosing a grout for injection. Therefore, the present research work used only composite grouts based on natural hydraulic lime and fly ash. According to the mentioned bibliography, a tentative to get an advantage of the beneficial effect of fly ash addition on the rheology of those grouts was made.

Taking into consideration all these points, several fly ash/binder ratios were tested (0%, 5% and 15% in weight) in order to understand their influence in natural hydraulic lime composites grout behaviour.

5.3.1.2 Material characteristics

The hydraulic lime used is a EN459-1 NHL5 produced in Portugal by Secil-Martingança, which has the characteristics presented in Table 5.1 according to the information of the quality control system provided by the manufacturer. Expansibility test for hydraulic lime was made according to EN 196-3.

Table 5.1: Natural hydraulic lime characteristics.

Compression resistance at 7 days (MPa)	**5,5**	
Fineness	90 μm	**11.90%**
	200μm	**1,6%**
Setting time	start	**2h45**
	end	**6h37**
Expansibility	**0,79mm**	
Free lime	**3,89%**	
SO_3	**3.23%**	
Ignition loss	**19.84%**	

Table 5.2 presents chemical characterizations of NHL5 according to SEM results:

Table 5.2: Chemical characterizations of NHL5 according to SEM results.

Formula	**(%)**
Na2O	0.96
MgO	0.78
Al2O3	1.43
SiO2	62.88
P2O5	1.77
K2O	0.52
CaO	3.78
TiO2	4.3
MnO	9.75
Fe2O3	12.54
ZrO2	1.29

The fly ash used is a silicious one (with pozzolanic activity), according to EN451 (Table 5.3).

Table 5.3: Chemical characterizations of fly ash according to SEM results.

Formula	**(%)**	**Formula**	**(%)**
CO_2	-	CaO	8.23
Ignition loss	2.34	TiO_2	1.37
Insoluble residue	66.96	Cr	0.02
Free lime	0.78	MnO	0.03
Chloride	-	Fe_2O_3	8.97
Na_2O	1.78	Co	-
MgO	1.74	Cu	0.01
Al_2O_3	21.86	Zn	0.02
SiO_2	48.75	As	0.01
P_2O_5	0.89	Sr	0.35
SO_3	1.79	Ba	0.23
K_2O	1.57	Pb	0.01

5.4 Temperature effect on grout rheological behaviour

A study was developed which aims at contributing to better understand the flow behaviour that natural hydraulic lime-fly ash based grouts presents under different temperatures. In order to do that, some rheological tests were done, such as in fly ash situation for fresh grout. The present work was designed so that the effects of different ratios of fly ash (0%, 5% and 15%) and different grout temperatures (5ºC, 20ºC and 35ºC) in some rheological parameters such as yield stress, plastic viscosity, power-law index and consistency could be better understood. Thixotropy is discussed in Chapter 6.

Several water/binder (w/b) ratios were used (0.60, 0.70 and 0.80) in order to understand their influence in natural hydraulic lime grout behaviour. The w/b range adopted was based in the bleeding results presented in Chapter 7 (Bras *et al.*, 2008). Fresh and hardened properties were tested. In this context, “fresh” means within the first 30 min after the binder first comes into contact with water.

The experimental program was conceived in accordance with the Taguchi method, so that the optimum working conditions of the factors which affect those fresh properties of natural hydraulic lime-fly ash grouts could be identified.

5.5 Taguchi method

5.5.1 Introduction

Considering the wide range of variables that may influence the grout behaviour, a previous definition of which of them should be used and which levels should be included was made, leading to a carefully conceived experimental campaign using Design of Experiments (DOE). DOE is a powerful statistical technique for improving product/process designs and solving production problems. A standardized version of the DOE, as forwarded by Dr. Genichi Taguchi, allows one to easily learn and apply the technique for product design optimization and production problem investigation.

The Taguchi approach of DOE was introduced to USA and other western countries only in the early 1980's and since then it has been a popular product and process improvement tool in the hands of the engineering and scientific professionals. For experiment designs, Taguchi created a set of tables of numbers known as orthogonal arrays (OA) designated as L-4, L-8, L-9, L-32, etc. They are used to lay out experiments of particular factor constituents. For analysis of results, he follows the basic statistical calculation such as average and analysis of variance (ANOVA), but blends with it a new approach to analyze results based on the deviation from the target instead of absolute values. The study of results based on the deviation from the target (or the average when a target is absent) allows selection of the design condition that is most consistent and yields reduced variation, which leads to improved quality (Roy, 2001) (Fig. 5.2).

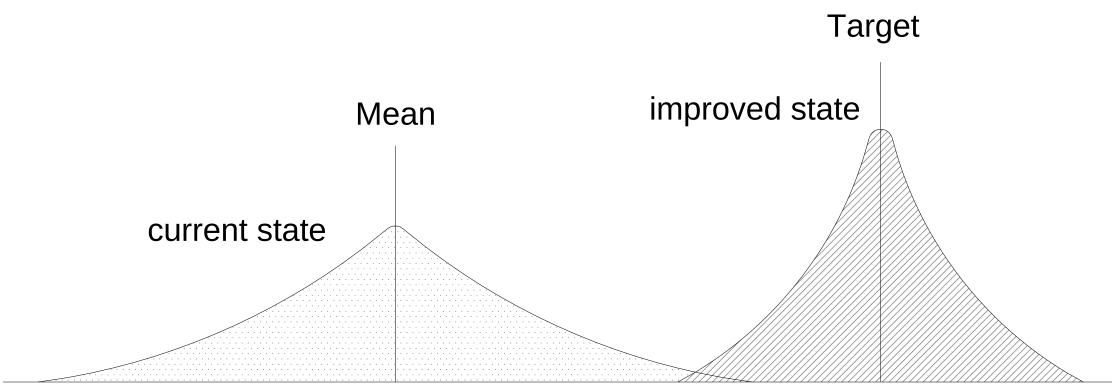

Figure 5.2: Improving quality by reducing variation around the target.

The use of the Taguchi's method allowed a considerable reduction of the number of experiments, when compared to the full factorial, therefore optimising the controllable inputs and measurable outputs.

An experiment with *f* control factors (variables) and *n* levels per variable would imply a full factorial of n^f; by using the Taguchi method this number can be considerably reduced, although maintaining the reliability of the campaign. This technique also enables the evaluation of the effects of each of the considered factor on the final results.

According to (Roy, 2001), factors are variables (or parameters) that have direct influence on the performance of the product or process under investigation. They are of two types:

Discrete - assumes known values or status for the level, such as: container, type of materials, etc.
Continuous - can assume any workable value for the factor levels, such as: temperature, pressure, thickness, etc.

Levels are the values or descriptions that define the condition of the factor held while performing the experiments.

To study influence of a factor, it is necessary to run experiments with two or more levels of the factors. Two is the minimum number of levels required to make comparison of the performance and thereby determine the influence. If the actual behaviour is non-linear it is required more than two levels per factor and at least, three levels are desirable. Nonlinearity dictates levels for continuous factors only.

The use of Taguchi's orthogonal arrays implies the knowledge of the number of experiments to be performed and the manner in which they should be carried out, i.e., number and the factor level combinations. Each array can be used to suit a number of experimental situations. To design experiments, Taguchi has offered a number of orthogonal arrays (OA): OA for 2-Level Factors; OA for 3-Level Factors and OA for 4-Level Factors. The smallest among the orthogonal array is an L-4 constructed to accommodate three two level factors.

In an array, numbers represent factor levels, rows represent trial conditions, columns accommodate factors, columns are orthogonal and each array is used for many experiments.

The standard notation for Orthogonal Arrays is:
Symbol L_n (X^Y)
Where n = Number of experiments

X = Number of levels

Y = Number of factors

Example:

L_8 (2^7),

where:

8 = Number of experiments

2 = Number of levels

7 = Number of factors

The method is to run the experiments by reading and using the level factors represented in each row of the OA. Simple analysis (using only arithmetic calculations) of results of the experiments can produce most of the useful information. Such calculation comprises the calculation of average effects of all factor levels and the grand average of the results.

Once all the average effects are calculated, for visual presentations and understanding, the factor average effects are graphed in a suitable scale. The spacing between the factor levels and between two factors, is arbitrary. However, the spacing between the two extreme levels of the factors should be maintained the same. For selecting the scale of the ordinate (y-axis) of the plot, note that the average effects of the two levels for two level factors are spaced equally above and below the grand average line. Such display of the average effects of factors indicates the trend of influence of the factors and is also known as main effect, column effect or factorial effect. That trend of influence indicates how does factor behaves, what is the factor influence in the variability of results, what is the condition more desirable and even how much funds can be saved.

To determine which among the levels of the factor is most desirable for achieving project objectives, it is necessary to establish the Quality Characteristic (QC) applicable. The QC applicable for the measure of the objective is a function of the measurement and its units used to evaluate how well the performance satisfies the objective.

Examples:

a) Nominal is Best: 3000 rpm, 2 volt battery, etc.

b) Smaller is Better: noise, bleeding, rejects, surface roughness, etc.

c) Bigger is Better: strength, efficiency, etc.

According to the average effects and based on the QC, the most desirable condition (Optimum condition) is determined. The estimate of the expected performance obtained carries a statistical meaning which needs to be understood before proceeding to confirm the prediction of the performance. The performance at the optimum condition, obviously, is expected to present an improvement over the current status (need to be assumed or determined). First, there is the need to interpret the meaning of the expected performance and then determine the improvement it potentially offers. Generally, the optimum condition will not be one that has already been tested. Thus, it will be necessary to run additional experiments to confirm the predicted performance.

Confidence Interval (C.I.) on the expected performance can be calculated from statistical calculations called ANOVA calculation. These boundary values are used to confirm the performance. ANOVA offers the following statistics:

a) Relative influence of factors and interactions;
b) A level of confidence on the estimated performance at optimum condition and main effects;
c) Significance of factor and interaction influence.

The technique for calculation of ANOVA statistics is too complex and is beyond the scope of this chapter. For detailed information see (Roy, 2001).

5.5.2 Grout composition optimization

In this study, three control factors were used: w/b ratio (A), fresh grout temperature (B) and fly ash percentage (C). For each factor three control levels were considered, as show in Table 5.4. An L_9 OA is appropriated for this purpose (Table 5.5). This means that only 9 runs are necessary to optimize the grouts (according to injectability principles). This reduction means that only 33% of a full factorial experimental test will be made, which represents a great advantage.

Table 5.4: Factors and levels for this study.

Factors and Levels for Case Study				
Factor		Level		
		1	2	3
A	W/B	0.60	0.70	0.80
B	Temperature (ºC)	5	20	35
C	Fly ash (%)	0	5	15

Table 5.5: Orthogonal array L_9 with factor assignment for the experiments.

L-9:				
Experiment Design with Four Three- Level Factors				
Experiment	Factor			
	A	B	C	D
1	1	1	1	0
2	1	2	2	0
3	1	3	3	0
4	2	1	2	0
5	2	2	3	0
6	2	3	1	0
7	3	1	3	0
8	3	2	1	0
9	3	3	2	0

An L_9 is an OA used for experiments with 4 three-level factors. Since in this test there are only 3 three-level factors, this is an appropriated array. The columns of OA correspond to the level of control factors and the orthogonal rows to each experimental run, that is, a specific set of factor levels to be tested. In order to ensure independence among the experimental observations, which are necessary to warrant the use of statistical methods in experimental design, the randomization for both the allocation of the experimental material and the order in which the individual runs of the experimental are to be performed is required.

After conducting the experiments (using three samples in each condition) the analysis of results were made using average values. In order to distinguish all factors that are statistically significant, ANOVA was applied to the three factors (w/b, temperature and fly ash).

Then, it was used a test of significance that compare the calculated facto F-ratio (from ANOVA table) with the calculated reference value. The purpose is just to distinguish the significant factors from the insignificant ones. Thus, using the F statistic test to each factor, for a confidence level of 95%, pooled factors were controlled. The process of ignoring a factor once it is deemed insignificant, called pooling, is done by combining the influence of the factor with that of the error term. In this way, if the presence of a pooled factor is detected this means that the factor is not relevant for the expected final result and for the optimum condition.

The value of F for each factor ($F_{FACTOR} = F_A$, F_B or F_C) is used for the significance test. The higher the F_{FACTOR}, the larger the significant influence of that control factor will be. In a practical point of view, the value of F statistic represents the ratio of variance explained by control factors to the unexplained variance by errors in the experiment (Ozcan Tan *et al.*, 2005), (Roy, 2001), (Yong-Huang Lin *et al.*, 2004).

5.6 Procedure

5.6.1 Mixing procedures

Before preparing the grouts the dry hydraulic lime and the fly ash were hand mixed with a trowel to avoid the formation of granules. The mixing procedure adopted was the same as in 4.3.3 and the mixture procedure adopted was the following (Bras *et al.*, 2009): the whole binder (natural hydraulic lime and fly ash) is added to 17/24 water and mixed during 2 minutes. The remaining water is added within 30 seconds without stopping the mixer. After all materials have been added, the mixture was maintained for 4 min at 2100 rpm.

Three tests were conducted for every mixing procedure. The relative humidity of the laboratory was 55% and the temperature 24±2 ºC.

5.6.2 Rheological measurements

The rheological properties of the different grouts were studied using a Bohlin Gemini HR_{nano} rotational rheometer, in a plate/plate geometry (ϕ = 40mm), with a gap of 2mm (which corresponds approximately to 10 times the maximum particle size). The samples were subjected to a pre-shearing stage during 30s at $\dot{\gamma} = 10s^{-1}$, 8 minutes after binder were placed in water. Once 60s at rest, a step up test of shear rate was applied including a linear 10-min upwards from 0 to 300s^{-1}, 60 s at maximum shear rate and an analogous step-down from 300s^{-1} to rest. The time step adopted was the necessary to obtain the steady state behaviour for each shear rate applied. Nine experimental conditions were tested (Table 5.6).

Table 5.6: Experimental conditions tested.

Test :		**1**	**Test :**		**6**
A	W/B	0.60	A	W/B	0.70
B	Temperature (ºC)	5	B	Temperature (ºC)	35
C	Fly ash (%)	0	C	Fly ash (%)	0
Test :		**2**	**Test :**		**7**
A	W/B	0.60	A	W/B	0.80
B	Temperature (ºC)	20	B	Temperature (ºC)	5
C	Fly ash (%)	5	C	Fly ash (%)	15
Test :		**3**	**Test :**		**8**
A	W/B	0.60	A	W/B	0.80
B	Temperature (ºC)	35	B	Temperature (ºC)	20
C	Fly ash (%)	15	C	Fly ash (%)	0
Test :		**4**	**Test :**		**9**
A	W/B	0.70	A	W/B	0.80
B	Temperature (ºC)	5	B	Temperature (ºC)	35
C	Fly ash (%)	5	C	Fly ash (%)	5
Test :		**5**			
A	W/B	0.70			
B	Temperature (ºC)	20			
C	Fly ash (%)	15			

5.6.3 Grout hardened properties tested

5.6.3.1 Strength, dynamic modulus of elasticity and carbonation test

Together with the previous tests, grout strength determination was made to the different experimental conditions tested (Test 1 to 9). In order to determine mechanical characteristics of the formulated grouts a testing campaign was undertaken and all samples were submitted to flexural and compressive strength tests following standard (NP EN 1015-11). The dynamic modulus of elasticity was determined following (Fe 08 DEC/UNL, 1996). Testing was performed at the ages of 28 days and the load was applied without shock at a uniform rate of 30 N/s for flexural strength. For compressive strength determination, the load was applied also without shock and was increased continuously at a rate of 300N/s. For both situations, the failure occurred within a period of 30s to 90s.

The grout carbonation depth was determined using a phenolphthalein pH-indicator (Fe 28 DEC/UNL, 1996).

5.6.3.2 Mercury intrusion porosimetry and grout shrinkage

In hydraulic based grouts such as cement, pore structure is a major component of the microstructure that affects water permeability and durability (Perraton *et al.*, 1988). Thus, for the optimal grout composition obtained according to the previous procedures, the pore size distribution of the hardened grouts and its changes due to time evolution was studied at age of 30 days and 120 days. Pore size distribution of the hardened grouts was determined using a Micromeritics' AutoPore IV 9500 Series mercury intrusion porosimetry. The experimental recommendations adopted were the ones used and suggested by Vasco Rato (Rato, 2006).

The pressure required to intrude mercury into the sample's pores is inversely proportional to the size of the pores. The instrument has a pressure capacity of 206 MPa (30 ksi) and is capable of penetrating pores as small as 7 nm in diameter (Fig. 5.3).

Knowing that the following procedure is not the best one (but the only available at that time), the fresh grouts tested were involved in plastic sheets immediately after its preparation in order to minimize hydraulic lime reactions with CO_2. The goal was to study, as much it was possible, the effect of fly ash addition in terms of porosity.

Figure 5.3: Porosimeter used in tests.

Grout shrinkage measurements were also performed since this phenomenon can lead to the formation of shrinkage cracks, which may affect the long-term performance of the grout. The method involves preparing prismatic specimens (4x4x16cm^3) that were removed from the molds at the age of 10h and storing in air for 90 days in ambient temperature. Length changes of the prisms were determined weekly using a length comparator.

5.7 Results and discussion

5.7.1 Rheological tests

The wide range of conditions tested in this work (for w/b, temperature and fly ash dosage) produced a variety of types of flow curves. For each test (from a total of 9) plots of variation of shear stress as a function of shear rate and apparent viscosity versus shear rate were analysed. Fig. 5.4 and 5.5 show the type of curves obtained with a grout with: w/b=0.70, 5% of fly ash, measured at a temperature of 5ºC. The curves obtained for the other grouts and temperatures are qualitatively similar.

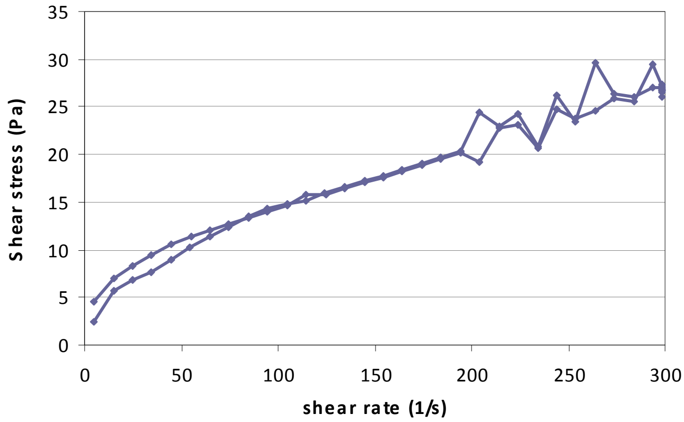

Figure 5.4: Shear stress *versus* shear rate for test 4 sample.

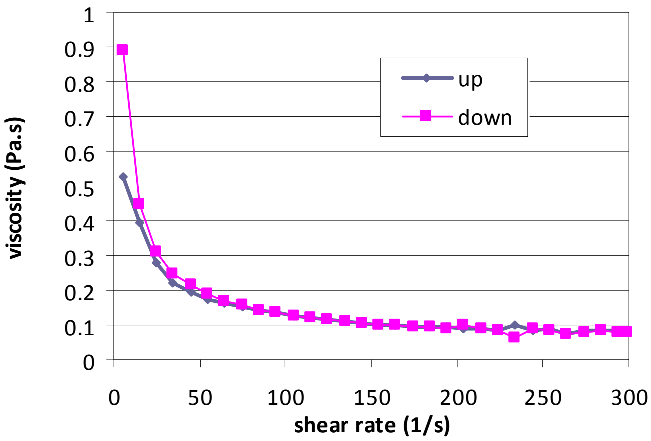

Figure 5.5: Apparent viscosity *versus* shear rate for test 4 sample.

There is a decrease of grout viscosity for all mixes when shear rate increases, showing a shear thinning behaviour for the hydraulic lime-fly ash grouts. Since all analysed fluids present a shear-thinning behaviour and knowing that it is possible to get η_∞ (second Newtonian region) for all mixtures, a Sisko model was adopted (Eqn. 5.1). In this way, K_2 (called the consistency ($Pa.s^n$)) and *n* (called the power-law index) were acquired.

In order to know the ability of the grout in filling up the voids and flowing when a shear stress is applied, the yield stress value (τ_0) for each sample was calculated. The same was made for plastic viscosity (η_p), using the modified Bingham equation.

The average values obtained for yield stress, plastic viscosity, consistency and power-law index are presented in Table 5.7, for each mixtures tested.

Table 5.7: Average values of rheological parameters for 9 mixtures.

Experiment	Yield stress (Pa)	Plastic viscosity (Pa.s)	K_2 ($Pa.s^n$)	*n*
1	14.49	0.86	13.10	0.20
2	9.92	0.72	9.00	0.24
3	6.11	0.55	6.20	0.21
4	2.27	0.17	1.53	0.31
5	0.44	0.12	0.33	0.53
6	5.41	0.29	5.70	0.11
7	1.38	0.11	0.77	0.39
8	2.34	0.14	1.70	0.27
9	1.73	0.19	1.40	0.34

In the figures below (Fig. 5.6, 5.7 5.8 and 5.9), the plot of the factor effects shows the trend of influence of the factor (w/b, temperature and fly ash) as well as their relative influence to the variation of the result, according to the Taguchi method.

Yield stress and plastic viscosity:

A smaller yield stress is better than a greater one. So, for yield stress results (Fig. 5.4), by comparing the slope alone, it is evident that w/b ratio is the most influential factor, since the average performance of all factors in result is 4.90 Pa and the amount of improvement obtainable by changing w/b level is greater. However, the decrease observed in yield stress by increasing the w/b ratio is less significant for w/b>0.70 (level 2). Fly ash has also an expressive effect in yield stress. More percentage of fly ash leads to an almost linear decrease of that result. Temperature effect is less significant. For fresh grout temperatures less than 20ºC there is a decrease of yield stress, however not very expressive. After that, it was not detected changes in result for T>20ºC.

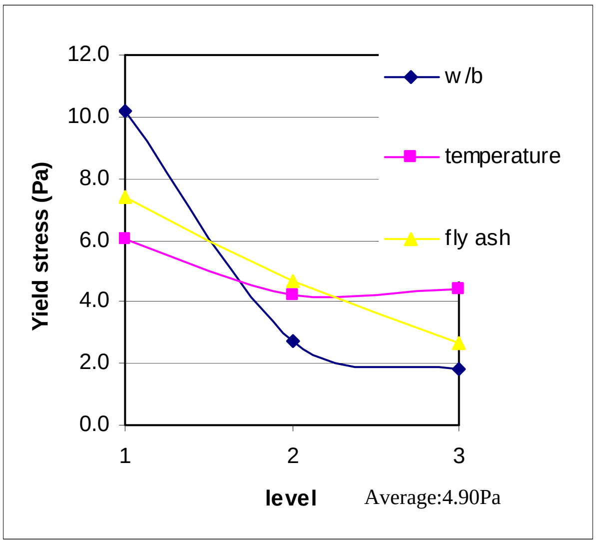

Figure 5.6: Influence of factor effects on yield stress results.

Since a smaller grout viscosity leads to a greater velocity, this is a desirable factor. In Fig. 5.7 it is evident that w/b ratio is again the most influential factor since the average performance of all factors in result is 0.35 Pa.s and the amount of improvement obtained by changing the w/b level is greater. However, the decrease observed in plastic viscosity by increasing the w/b ratio is less significant for w/b>0.70 (level 2). Fly ash has also an expressive effect in plastic viscosity. More percentage of fly ash leads to a linear decrease of plastic viscosity. The effect of temperature is less significant and was detected as being a pooled factor in this case (for a confidence level of 95%). It means that temperature is not very relevant to the final results, which shows that in the range $5°C \leq T \leq 35°C$ plastic viscosity is not dependent on temperature.

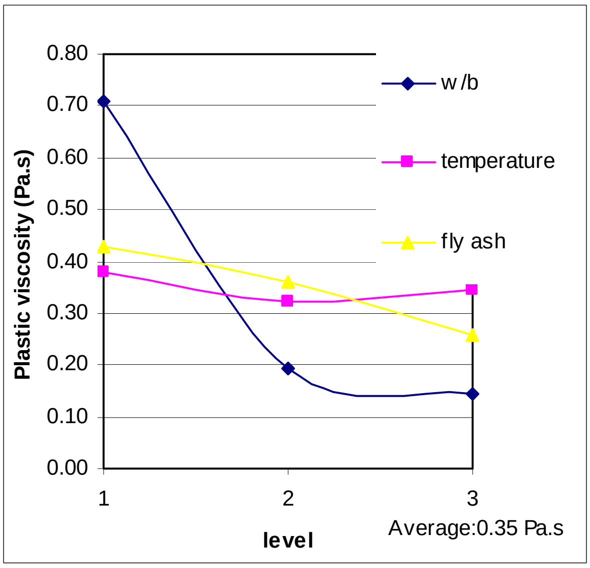

Figure 5.7: Influence of factor effects on plastic viscosity results.

Fresh grout properties like yield stress and plastic viscosity decrease with an increased percentage of water in grout composition. However, the decrease observed in yield stress and plastic viscosity by increasing the w/b ratio is less significant for w/b>0.70. That fact leads to an improvement of injectability and grout fluidity. A draw-back of increasing w/b is the fact that stability and mechanical strength problems may come out. Nevertheless, it does not seem the case even for w/b equal to 0.80, since bleeding does not exceed 2.5%, according to experimental results obtained before.

The introduction of fly ash in the grout composition leads to a decrease of yield stress that can be detected with only 5% of fly ash. Between the range 5% to 15% this decrease is practically linear, with a slope slightly higher between 0 and 5% than between 5% and 15%. As it was mentioned before by Mohammed Sonebi (Sonebi, 2006), a reduction of yield stress value was detected when cement was replaced by pulverised fly ash in cement based grouts, due to ball bearing effect.

The effect of temperature seems to represent the least significant factor for yield stress results. Our results (Fig. 5.6) seem to show that in the range between 20ºC and 35ºC yield stress is almost constant, which is the opposite of what was expected. In fact, at high temperatures it was expected an increase of microstructure density which could increase yield stress. Considering the very limited bibliography concerning hydration of hydraulic lime during the first moments after mixing with water, it was focused only the description of the experimental findings.

For plastic viscosity variation (Fig. 5.7), as it was observed, fresh grout temperature does not have a significant effect on that type of result. Taking into consideration that only fresh grout is able to be injectable, for grout temperatures ranging between 5ºC and 35ºC it seems that the velocity at which a given grout will flow, after the beginning of flow, is independent of temperature.

Consistency and power-law index:

Since an increasing of the K_2 parameter leads to a grout viscosity increase, a smaller result is more desirable. According to Fig. 5.8 it is evident that the w/b ratio is again the most influential factor, since the average performance of all factors in result is 4.42 $Pa.s^n$ and the amount of improvement obtainable by changing w/b level is greater. However, the decrease observed in consistency by increasing the w/b ratio is less significant for w/b>0.70 (level 2). Fly ash has also an expressive effect. More percentage of fly ash leads to a linear decrease of K_2. As in the yield stress case, the addition of only 5% of fly ash leads to a decrease of consistency, with a slope slightly higher between 0 and 5% than between 5% and 15%.

Temperature effect is less significant and was detected as being a pooled factor in this case (for a confidence level of 95%).

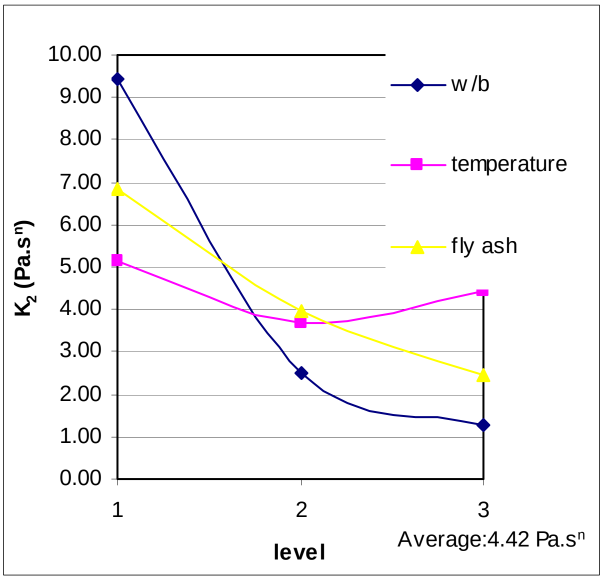

Figure 5.8: Influence of factor effects on consistency results.

The previous results show that the grout compositions tested presented a shear-thinning behaviour, which means that *n* must be less than 1. Since a pseudoplastic behaviour means that if flow velocity decreases during grout injection it leads to a viscosity increase (which is not desirable), *n* value should be closer to 1 (a bigger result is better). According to Fig. 5.9, the most influential factor is fly ash. A bigger fly ash ratio leads to an almost linear increase of *n*, which means that level 3 (%fly ash= 15%) leads to a less pseudoplastic grout. Once again the addition of only 5% of fly ash leads to an increase of *n*. For the w/b ratio there is an expressive increase of *n* between $0.60 \leq w/b \leq 0.70$, after that a light increase is detected until w/b=0.80. Grout temperature increases *n* value until T=20ºC is reached, after which it can be observed a significant decrease of *n*. No pooled factors were detected for a confidence level of 95%.

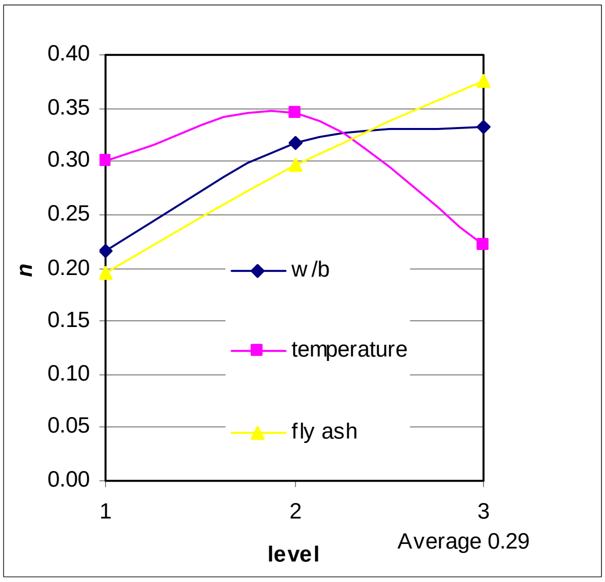

Figure 5.9: Influence of factor effects on power-law index results.

Baronio and co-workers (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 properly. During injection, when the grout reaches a large void, no pressure can be built up in the neighbourhood of that void. Due to this low pressure, the grout will enter the fine cracks only over a short distance. When the large void is finally filled, the pressure can increase again but too much water of the grout is absorbed in the fine cracks to restart the flow. The zone hidden by the finer cracks will never be injected through a single hole. An approximation to a Newtonian behaviour enables that grout to flow easily inside porous medium, even when velocity decreases during injection, since viscosity will not change too much.

The effect of fly ash on n could be explained by the spherical shape and the small size of fly ash, compared with natural hydraulic lime (particles with elongated shape), as it can be observed in Fig. 5.10 and 5.11.

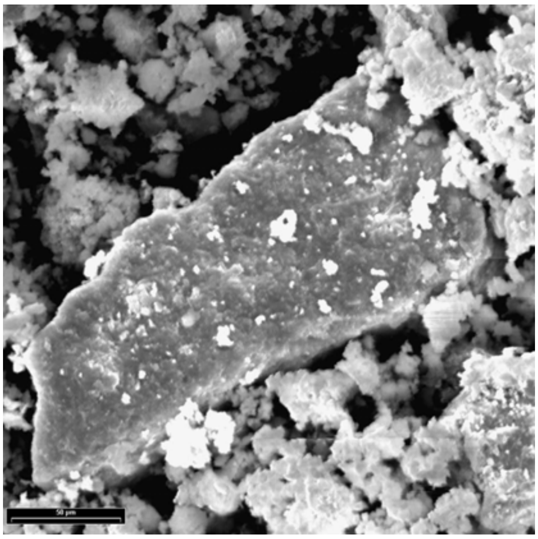

Figure 5.10: SEM image of natural hydraulic lime particle at 1500x.

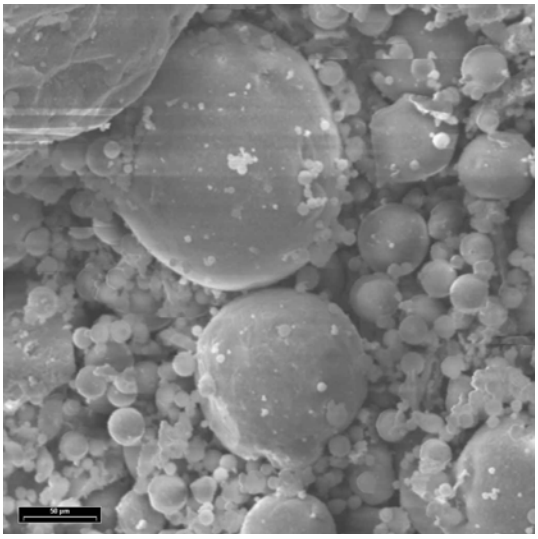

Figure 5.11: SEM image of fly ash particle at 1000x.

With the increasing shear rate the elongated particles tend to a preferential orientation inside the fresh grout when flow occurs. In this way, there is a viscosity decrease, which explains the previous results obtained (Bras *et al.*, 2009). The spherical shape of fly ash enables an almost Newtonian behaviour of these grouts, because a parcel of natural hydraulic lime (particles with elongated shape who lead to a shear-thinning behaviour) is replaced by fly ash.

Several grout compositions were tested and rheological analyses were made in order to know the effect of some grout components in the fresh grout properties. Different w/b ratios were tested (0.60, 0.70 and 0.80), as well as different fly ash ratios added to natural hydraulic lime (0%, 5% and 15%) and different grout temperature (5ºC, 20ºC and 35ºC). Four types of rheological parameters were obtained: yield stress, plastic viscosity, consistency and power-law index. The individual effect of each factor on results was observed and an optimal grout composition was reached. According to the levels and factors studied, the optimum solution for a better grout injection capacity leads to a natural hydraulic lime based grout with w/b=0.80, a fly ash ratio of 15% and a fresh grout temperature of 20ºC (Table 5.8). Using F statistic test for a confidence level of 95%, a confidence interval for each rheological value was obtained (Table 5.9).

Table 5.8: Optimum natural hydraulic lime- fly ash grout composition.

w/b	0.80
Temperature (ºC)	20
Fly ash (%)	15

Table 5.9: Expected results at optimum grout composition (for a confidence level of 95%).

	yield stress (Pa)	plastic viscosity (Pa.s)	consistency ($Pa.s^n$)	power-law index
low value	-2.37	-0.05	-1.85	0.40
high value	0.16	0.15	0.46	0.56

It is important to point out that negative values of any of the above parameters lack any physical meaning, which means that the real intervals that must be considered starts at a zero value.

Since the mean of samples tested at the optimum condition (the experimental confirmation of the predicted results) was shown to be within the confidence interval (yield stress: -0.24MPa, plastic viscosity: 0.12Pa.s, consistency: 0.20$Pa.s^n$, power-law index: 0.48), it means that the results are within the predicted performance even in the presence of interactions. Consequently, it means that the interactions between factors are small and/or they nullify each other resulting in a total effect that is small.

Real masonry is heterogeneous and has always zones with very different porosity. Besides that, the coarse or the fine zone is not always the most endanger one. Thus, the grout criteria selection should always take also into account this important variable, knowing that there are always limitations in the methodology. Laboratory injectability tests have been made by the authors. Those studies are not shown in the present work in spite of its relevance for injectability purposes; although it will be considered in future developments (see Chapter 7).

5.7.2 Strength, dynamic modulus of elasticity and carbonation

The flexural and compressive strength of the previous grouts were tested at 7 days and 28 days, for three different w/b adopted: 0.60, 0.70 and 0.80 and three different fly ash content (0%, 5% and 15%). The grout temperature stayed constant during all process (T=25ºC). The average measurements of flexural and compressive strength are presented together with its coefficients of variation (CV) in Table 5.10 and 5.11.

Table 5.10: Strength results for different procedure for NHL5 based grout with or without fly ash at 7days (temperature=25ºC).

T=25ºC	Average strength at 7 days			
	Flexural (MPa)	CV (%)	Compressive (MPa)	CV (%)
Test 1	0.8	28.1	4.4	5.0
Test 2	0.5	20.9	4.7	1.8
Test 3	0.9	14.8	3.4	1.2
Test 4	0.7	23.5	2.6	11.6
Test 5	1.0	18.6	2.9	5.7
Test 6	0.8	34.3	2.6	4.7
Test 7	0.5	10.5	1.3	13.6
Test 8	0.6	5.4	1.8	1.2
Test 9	0.6	13.4	1.7	10.1

Table 5.11: Strength results for different procedure for NHL5 based grout with or without fly ash at 28 days (temperature=25ºC).

T=25ºC	Average strength at 28 days			
	Flexural (MPa)	CV (%)	Compressive (MPa)	CV (%)
Test 1	0.2	35.3	8.2	7.9
Test 2	0.1	36.1	6.1	2.0
Test 3	2.1	2.3	7.0	3.0
Test 4	1.4	0.0	5.7	6.9
Test 5	1.4	31.6	3.2	1.1
Test 6	1.7	19.8	5.8	8.2
Test 7	0.8	27.4	3.3	0.8
Test 8	0.1	10.4	3.6	9.5
Test 9	0.0	30.6	3.4	5.9

Table 5.10 and 5.11 shows that there are some differences in flexural strength results and some dispersion may be found, especially for flexural than for compressive strength. Flexural strength results at 7 days goes from 0.5MPa (grout with w/b=0.80 and 0% of fly ash) to 1.0MPa (grout with w/b=0.70 fly ash=15%). At 28 days an increase of strength is detected especially for Test 5 and 6 (grout with w/b=0.70 fly ash=15%) and (grout with w/b=0.70 fly ash=0%) respectively.

In the figures below (Fig. 5.12, 5.13, 5.14 and 5.15), the plot of the factor effects for 7 days and 28 day shows the trend of influence of the factor (w/b and fly ash) as well as their relative influence to the variation of the flexural strength results, according to the Taguchi method (see also Table 5.4).

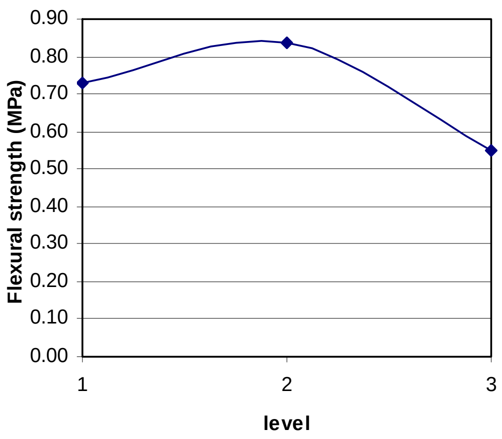

Figure 5.12: Influence of w/b effects on grout flexural strength results at 7 days.

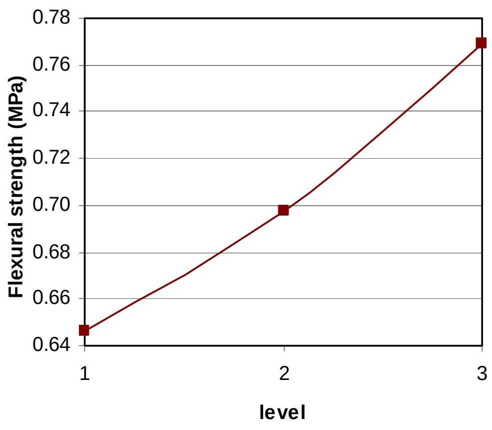

Figure 5.13: Influence of fly ash content on grout flexural strength results at 7 days.

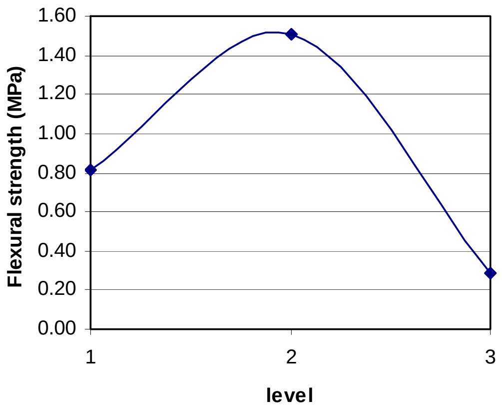

Figure 5.14: Influence of w/b effects on grout flexural strength results at 28 days.

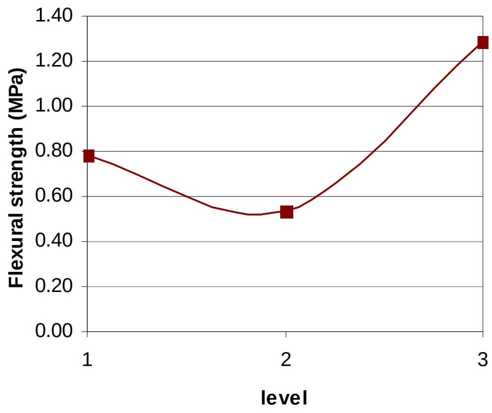

Figure 5.15: Influence of fly ash content on grout flexural strength results at 28 days.

According to the previous results, a grout with w/b=0.70 together with a fly ash content = 15% leads to the highest flexural strength. In fact, it was also detected an expressive increase of flexural strength due to fly ash (Fig. 5.13 and 5.15) probably due to pozzolanic reactions, especially if fly ash is increased from 5% to 15%. The development of strength at the beginning is due to and determined by the hydraulic lime content. This is not surprising, because the hydraulic activity of the pozzolans (like fly ash) does not start early and is a slow process as it was observed by Paulina Faria (Faria, 2004). Four weeks seem to be enough to detect those changes.

Trend of influence of the factor (w/b and fly ash) as well as their relative influence to the variation of the compressive strength results for 7 days and 28 days is shown in Fig. 5.16, 5.17, 5.18 and 5.19.

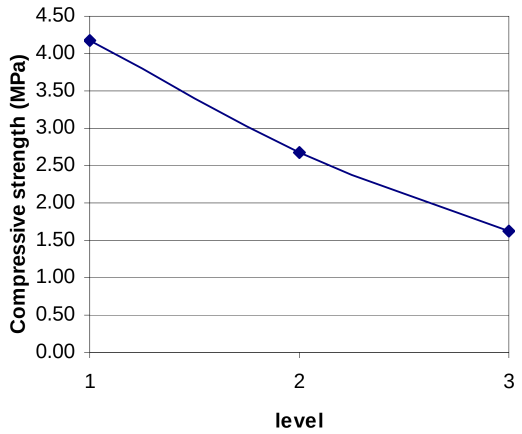

Figure 5.16: Influence of w/b effects on grout compressive strength results at 7 days.

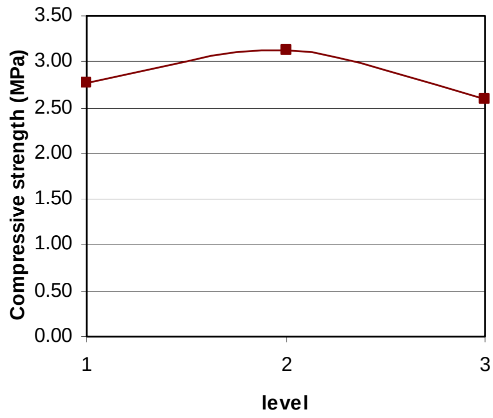

Figure 5.17: Influence of fly ash content on grout compressive strength results at 7 days.

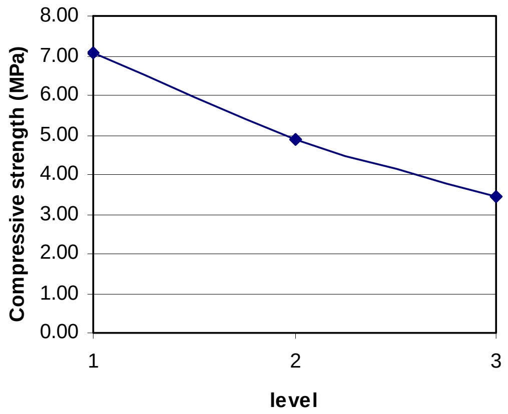

Figure 5.18: Influence of w/b effects on grout compressive strength results at 28 days.

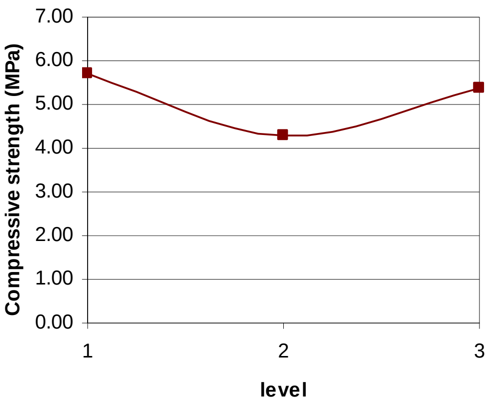

Figure 5.19: Influence of fly ash content on grout compressive strength results at 28 days.

Water binder content seems to decrease compressive strength. However, the introduction of fly ash does not lead to the same behaviour. At 7 days there is a slightly strength increase until 5% of fly ash and after that, it starts to decrease. The analysis of the previous figures show that in a general way, compressive values are higher 4 weeks later and 15% of fly ash lead to best results, also probably due to pozzolanic reactions.

From rheological point of view, the optimum solution achieved for grouts made of fly ash and natural hydraulic lime is the following: w/b=0.80; temperature=20ºC and fly ash=15%. However, since only small differences were observed when the w/b ratio increases from 0.70 to 0.80, it seems probable that the optimum solution range could be extended to a w/b=0.70, therefore increasing rheological and mechanical properties. Thus, taking into consideration rheological and strength results it seems that the best solution is the grout composition with w/b=0.70 (level 2) together with a fly ash content= 15% (level 3). For this grout composition dynamic modulus of elasticity and carbonation depth were measured for 7 and 28 days of age (Table 5.12).

Table 5.12: Dynamic elastic modulus and carbonation depth for the optimum procedure for NHL5 based grout with 15% of fly ash at 7 and 28 days of age (temperature=25ºC).

E_{7d} (MPa)	CV (%)	E_{28d} (MPa)	CV (%)	Carbonation $depth_{7d}$ (mm)	CV (%)	Carbonation $depth_{28d}$ (mm)	CV (%)
2195.3	5.0	2859.3 MPa	12.0 %	5.0 mm	0.0 %	16.0 mm	0.0 %

As it may be observed even elastic modulus increase with time, going from 2195 MPa to 2859 MPa, which represent an increase of 30%. Carbonation depth became higher with age. In a first approach this previous phenomenon could be explained by a microstructure weakness. However, mechanical strength shows the opposite, meaning that its microstructure is stronger. To get more information concerning microstructure, mercury intrusion porosimetry was used.

5.7.3 Mercury intrusion porosimetry and grout shrinkage

For the optimal grout composition obtained according to the previous procedures, it was studied the pore size distribution of the hardened grouts with NHL5, 15% of fly ash, w/b=0.70 and its changes due to time evolution at age of 30 days and 120 days.

In a cement paste, it is suggested that capillary pores larger than 10 nm influence mostly the strength and permeability; while gel pores smaller than 10 nm influence the drying shrinkage and creep (Mehta, 1986). The capillary pores can further be divided into large (>50 nm) and medium (50–10 nm) capillary pores. The same assumption was used for NHL5 studies.

The grouts tested were involved in plastic sheets immediately after its preparation in order to avoid hydraulic lime reactions with CO_2. The goal was to study the effect of fly ash addition in terms of porosity. Fig. 5.20 shows the volume of pores in the three size categories for samples cured for 30 days and 120 days. This information was determined using mercury intrusion porosimetry.

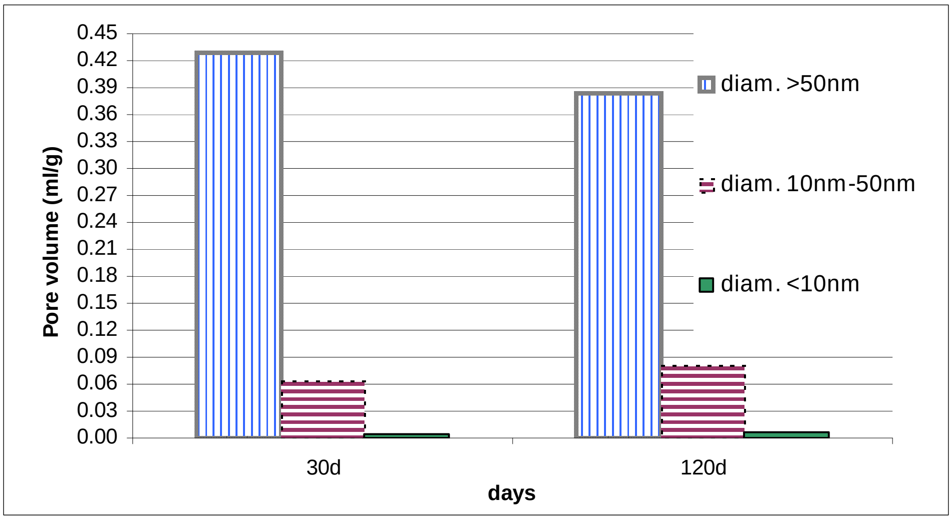

Figure 5.20: Volume of pores in the three size categories for samples cured for 30 days and 120 days, for NHL5 based grout with 15% of fly ash.

As it can be observed, there is a reduction of the total porosity of the grout with time evolution. For a grout with 30 days its porosity is 54.8% and for 120 days its porosity decrease to 52.6%. There is also a reduction of the higher size category of voids diameter, which represent a good result since strength will be improved (as it was previously confirmed) and reduce permeability. The pores smaller than 10 nm have double its volume (0.0033 ml/g at 30days and 0.0066 ml/g at 120 days) but when comparing to the total pore volume it does not seems to have an expressive relevance.

Grout cumulative shrinkage (autogenous and drying shrinkage) was measured for the two best compositions: NHL5 and NHL5+15%fly ash, with w/b=0.70 (Fig. 5.21). Fly ash represents a good option to minimize shrinkage. It can be observed that the shrinkage is smaller for the grout with fly ash (it is reduced almost 0.5mm/m) which is in agreement with (Mirza *et al.*, 2002).

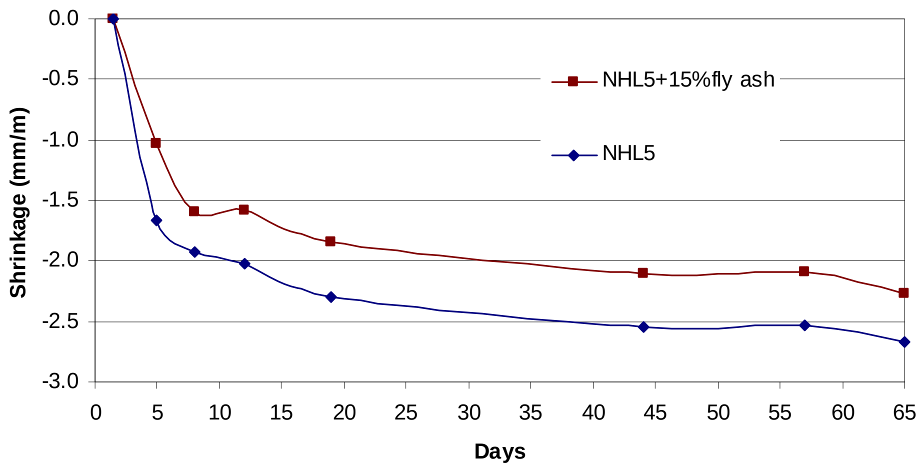

Figure 5.21: Cumulative shrinkage of NHL5 and NHL5+15% fly ash based grout (with w/b=0.70) from day 0 to 65.

5.8. Main conclusions

The first step for the application of injection technique in masonry consolidation includes the selection of grout composition and the control of masonry existing materials. For that, laboratory tests are carried out. At this stage grout injectability improvement must be checked by means of specific grout composition (selecting the optimal composition by DOE technique), which also provides the preliminary indications on the possibility of grout injectability in situ.

This chapter analyses some relevant rheological properties for natural hydraulic lime grouts, such as yield stress, plastic viscosity, consistency and power-law index. The study has envisaged to increase the understanding of the influence of environmental temperature in fresh grout behaviour and the influence of finer powders like fly ash in composite grouts for injection purpose. An effort to get an optimal solution was made using the Taguchi method.

In general terms the w/b factor is the most important. An increase of water/binder ratio leads to better rheological parameters: smaller yield stress, plastic viscosity, consistency and a higher power-law index. However, that improvement becomes less visible for w/b>0.70.
For the *n* value, fly ash is the most important factor. An increase of fly ash leads to an almost Newtonian grout behaviour. For yield stress and consistency values, simple addictions of 5% of fly ash significantly change those rheological parameters.

The effect of grout temperature in the analysed range ($5°C \leq T \leq 35°C$) is the least significant of the considered factors – only for the rheological parameters tested. If the environmental temperature is similar to the one of the fresh grout, probably the rheological parameters will not change substantially even if consolidation work starts early in the morning and ends at higher temperatures.

According to the previous results, w/b=0.70 together with a fly ash content= 15% lead to the highest flexural strength. In fact, it was also detected an expressive increase of flexural strength due to fly ash probably due to pozzolanic reactions, especially if fly ash is increased from 5% to 15%. Four weeks seem to be enough to detect those changes. Traditional masonry present very low tensile strength, so the increasing of grout tensile strength is quite important.

Compressive strength values are higher and present a trend line for time evolution similar to flexural behaviour. However, the dispersion of results (represented by CV) are higher for flexural than for compressive strength.

Taking into consideration rheological and strength results it seems that the best solution is the grout composition with w/b=0.70 (level 2) together with a fly ash content= 15% (level 3).

As it can be observed, there is a reduction of the total porosity of the grout with time evolution. There is also a reduction of the higher size category of voids diameter, which is related with a strength improvement (as it was previously confirmed) and reduce permeability. A limited shrinkage of NHL5 + 15% of fly ash grout was observed and it was reduced almost 0.5mm/m when compared with a NHL5 grout. In a durability point of view, those results are also good since it reduces the probability of micro-cracking along the interfaces between grout and the original materials of the wall.

Taguchi approach of DOE method enables an improvement of those grout properties.

5.9. References

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Chapter 6. Effect of environmental temperature and fly ash addition in hydraulic lime grout behaviour-shear and time dependence

6.1 Purpose

Trying to extrapolate the behaviour of cement grout to what happens in hydraulic materials chemistry, such as natural hydraulic lime, a study was developed which aims at contributing to better understand the flow behaviour that natural hydraulic lime-fly ash grouts presents under different temperatures. The rheological measurement designed in this work aims at establishing a determination of the thixotropy of each grout and thus compare how structured are the mixes in relation to their variables of preparation and test conditions. An attempt to find the temperature limit (T_{limit}) that isolates thixotropy effect from hydration reactions in a grout was made. For NHL5 based grout T_{limit} =20ºC and for NHL5+15% fly ash based grout T_{limit} =15ºC. Grouts characterization based on maximum resisting time, rate of flocculation and analysis of the hydraulic lime grout behaviour tested at different shear rates, was performed using a shear thinning model and assuming that the structure is shear and time dependent.

The goal is to use this methodology during mix proportioning and design for masonry injection purpose, for raw materials evaluation or even in quality control *in situ*. The characterization at both steady and transient states enables that information to be obtained and allows a decision process. The tested grout compositions were optimized compositions obtained using design of experiments method (see Chapter 5). Two compositions were tested: hydraulic lime grout with water/binder (w/b) content equal to 70% without and with 15% of fly ash, respectively.

6.2 Introduction

Grout flow properties are affected by a large number of parameters (Eriksson *et al.*, 2004), (Fernàndez-Altable *et al.*, 2006), (Bras *et al.*, 2009). According to (Bras *et al.*, 2009) it is known that hydraulic lime based grout (with or without addition of fly ash) presents a shear thinning behaviour which means a viscosity decrease with an increase in shear rate. Since those grouts have a specific a yield stress value, they only will flow when shear stress exceeds the yield point. Those materials are called viscoplastic and they behave as a solid below the yield stress value and as a liquid above that. The flow curve associated to this behaviour is non-linear (Fig. 3.4 and 3.5).

The viscoplastic behaviour is found in flocculated suspensions, like cement or hydraulic lime grout, mortar and concrete (Wallevik J., 2009), (Hackley *et al.*, 2001), (Wallevik, 2009). They are dispersions where the dispersed particles form a network which is responsible for the yield point. The stronger the network is, the larger the stress necessary to overcome the internal structure. If the stress applied is lower than the yield value, it will cause an elastic deformation of the sample and its shape will be recovered. On the contrary, if the shear stress is higher than the yield stress (also larger than the internal network force to resist breakage of structure) then it will result in a continuous flow.

Generally, the behaviour of hydraulic lime based grout can be compared to a shear-thinning fluid as it occurs in cement grouts with low w/b ratios (similar to those used in this research work) (Bras *et al.*, 2009), (Rosquoët *et al.*, 2003). To describe the shear-thinning behaviour of those grouts usually it is used a model which combines the exponential behaviour with yield value – the Herschel-Buckley equation:

$$\tau = \tau_0 + A\dot{\gamma}^n \qquad (6.1)$$

where A and n are characteristic parameters describing the shear rate dependency of the material tested. When n>1 the equation signifies shear-thickening behaviour and n<1 means shear-thinning behaviour. This model, however, does not suit satisfactorily rheological behaviours of our tested grouts, not showing any clear yield stress (which is not true).

A comparison between measurements and literature (Wallevik, 2009), (Rosquoët *et al.*, 2003), (Barnes *et al.,* 2001), (Yahia *et al.*, 2001), finally allows for the selection of models for grout steady state description (Eqn 5.1 to 5.4).

However, that shear-thinning behaviour may not occur simultaneously but under a measurable length of time causing the material to be time dependent (Barnes *et al.,* 2001), (Barnes, 1997). The Non-Newtonian time dependent fluids are the fluids for which the viscosity depends not only on the shear rate applied to the sample but also on the time for which the fluid has been subjected to shearing. This can be expressed as

$$\eta = \eta(\dot{\gamma}, t) \tag{6.2}$$

These types of materials change their structure with the time of shear at a constant shear rate, which affects the viscosity even for that situation. Cementitious materials generally show a thixotropic behaviour (Fernàndez-Altable *et al.*, 2006), (Roussel *et al.*, 2007), (Roussel, 2006).

During the breakdown of the molecules/particles structure they start to orient themselves in the line of flow which reduces viscosity. It will take a certain time to break down the agglomerate into particles and to reach an equilibrium stress- the steady state. According to Roussel (Roussel, 2006), (Roussel, 2005), as long as steady state flow is reached, the behaviour of cement pastes may be described using a yield stress model such as the Bingham or Hershel Bulkley models. However, between two successive steady states, there is a transient regime, during which a yield stress model is not sufficient to describe the observed behaviour.

There are some models to describe the competition between breakdown and structural build-up (Roussel, 2006), (Coussot *et al.*, 2002), (Cheng *et al.*, 1965), among others (Eqn. 3.11 to 3.17).

Engineering consequences

From a rheological point of view, the goal is to design an homogeneous grout that is easily placed at the lowest possible cost. There are many ways to apply rheology in grouts technology. It is clear, however, that additional characterization of the flow and injection processes are needed, due to the complex composition of grouts and the complex interaction of flows and flow geometries which may evolve in time.

In situ grout application may occur at several environmental conditions, which may lead to different grout injection capacities, as reported by Eriksson *et a*l (Eriksson *et al.*, 2004). These authors noted the influence of water temperature as a factor able to influence grout properties. Other researchers have analysed the same influence, as it is the case of Fernàndez-Artable *et al* (Fernàndez-Altable *et al.*, 2006) that identified a relation between the grout rheological behaviour and the environmental temperature.

In concrete context, Roussel and Billberg (Roussel, 2006), (Billberg, 2006) stated that rheological phenomenon such as thixotropy plays an important role in the values of lateral stresses in the formwork when casting self compacted concrete (SCC) (see Chapter 3). In a similar way to what happens in SCC application, grout injection for masonry consolidation may lead to an increase of hydrostatic pressure and lead to structural damage. This means that thixotropic effect become self evident in grout design. Because the material builds up an internal structure, the lateral stress in masonry decreases quickly in the part of the injected grout that is at rest inside the porous media. Acording to Ovarlez *et al.* (Ovarlez *et al.*, 2006), this property enables the grout to withstand the load from grout injected above it, minimizing the increase of stress in the wall. This problem is associated to the flocculation rate of the grout since if it flocculates too much and the apparent yield stress increases above a critical value, the two adjacent grout layers do not mix at all and this probably will create a weak interface in the final structure.

Thus, a study was developed which aims at contributing to better understand the flow behaviour that natural hydraulic lime-fly ash grouts presents under different temperatures. The measurement programmes designed in this work aims at establishing a determination of the thixotropy of each grout and thus compare how structured are the mixes in relation to their variables of preparation and test conditions. An attempt to find the temperature limit that isolates thixotropy effect from hydration reactions in a grout was made. Maximum resisting time, rate of flocculation and analysis of the hydraulic lime grout behaviour tested at different shear rates was performed.

The grout compositions tested were the optimized compositions obtained in previous works using design of experiments method (DOE). Two compositions were tested: hydraulic lime grout with water/binder (w/b) content equal to 70% without and with 15% of fly ash, respectively.

6.3 Material characteristics

The selection of a binder to be used in grouts for injection should take into account the compatibility with the original material to be injected (Valluzzi, 2005). Thus, it was adopted a hydraulic lime EN459-1 NHL5 produced in Portugal by Secil-Martingança, which has the characteristics presented in table 6.1 according to the information of the quality control system provided by the manufacturer. Expansibility test for hydraulic lime was made according to EN 196-3. The fly ash used is a siliceous one (with pozzolanic activity), according to EN451 and its chemical characterization is in Table 5.3.

Table 6.1: Natural hydraulic lime characteristics.

Compression resistance at 7 days (MPa)	**5,5**	
Fineness	≥90 µm	**24.8%**
	≤200µm	**2.9%**
Setting time	start	**2h45**
	end	**6h37**
Expansibility	**0,79mm**	
Free lime	**3,89%**	
SO_3	**3.23%**	
Ignition loss	**19.84%**	

The grain size distribution of the binders is represented in the Fig. 6.1.

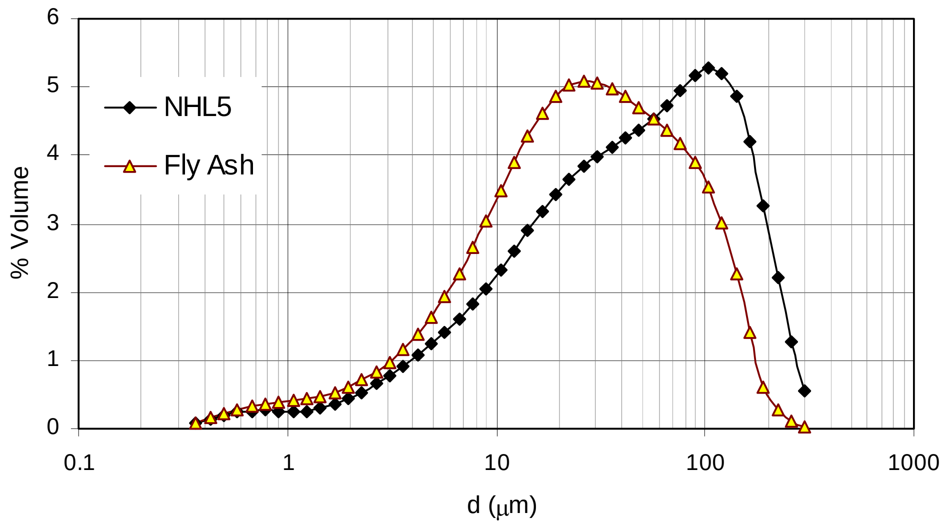

Figure 6.1: Grain size distribution (in volume) of NHL5 and fly ash adopted in the tests.

Density and fineness using Blaine permeameter of NHL5 and fly ash are represented in Table 6.2.

Table 6.2: Density and fineness using Blaine permeameter of NHL5 and fly ash.

Sample	Density (kg/m^3)	Fineness (Blaine) (m^2/kg)
NHL5	2850	578
Fly ash	2310	342

6.4 Procedure

6.4.1 Mixing procedures

Before preparing the grouts, the dry hydraulic lime was hand mixed with a trowel to avoid the formation of granules. The mixer blade had a helicoidal shape, with a diameter a little smaller than the cup diameter in order to allow all the grout to be mixed. The gap at the bottom, between the blade and the cup was 4mm. Ordinary tap water was used for the preparation of the grouts. The water was allowed to flow freely until a stable water temperature was reached.

Several mixing procedures were tested in order to understand their influence in natural hydraulic lime grout properties such as: the influence in grout water retention capacity, the influence in coagulation, among others. It was shown that there are differences in the grout behaviour when different mixing procedures are used and that fresh grout properties may be optimized if a proper mixing procedure is chosen (Bras *et al.*, 2009). The adopted mixing procedure was the following: the whole binder (natural hydraulic lime and fly ash) is added to 17/24 water and mixed during 2 minutes. The remaining water is added within 30 seconds without stopping the mixer. After all materials have been added, the mixture was maintained for 4 min at 2100 rpm. The w/b (in weight) studied was of 70%.

For each test, three samples were taken for every procedure. The relative humidity of the laboratory was 55% and the temperature 20±2 ºC.

6.4.2 Rheological measurements- time independent properties and temperature effect

The rheological properties of the different grouts were studied using a Bohlin Gemini HRnano rotational rheometer, in a plate/plate geometry ($\phi = 40mm$), with a gap of 2mm. The samples were subjected to a pre-shearing stage during 30s at $\dot{\gamma} = 10s^{-1}$, 8 minutes after binder were placed in water. Once 60s at rest, a step up test of shear rate was applied including a linear 10-min upwards from 0 to 300s^{-1}, 60 s at maximum shear rate and an analogous step-down from 300 s^{-1} to rest. The time step adopted was the necessary to obtain the steady state behaviour for each shear rate applied. For each grout sample different temperatures were applied: 5,10,15,20, 25 and 35ºC for NHL5 based grouts; 5 ºC, 10 ºC, 12.5 ºC, 15 ºC, 17.5 ºC and 20ºC for NHL5+15% fly ash based grouts.

To describe the grout rheological behaviour of hydraulic lime with fly ash based grouts it was chosen to adopt the Sisko model and the modified Bingham equation (Eqn. 6.2 and 6.5), since it suits better in this shear-thinning behaviour.

6.4.3 Rheological measurements- time dependent properties

Thixotropic materials, such as hydraulic lime based grouts, show a shear-thinning and time dependent behaviour. As the suspension is sheared, the weak physical bonds among particles are ruptured and the network among them breaks down into separate agglomerates, which can disintegrate further into smaller flocs (structural breakdown). If the suspension is at rest the particles will start to flocculate into agglomerates again (structural build-up). Thus, to study the time effect on this materials two procedures (1 and 2) where adopted in this study. In all of them, grout samples were subjected to a pre-shearing stage during 60s at $\dot{\gamma} = 300s^{-1}$, starting 8 minutes after binder were placed in water and mixed as it was explained before.

6.4.3.1 *Procedure 1*

Procedure 1 (Fig. 6.2): After the pre-shear, different resting times were applied to the sample: 30s, 60s, 100s, 200s, 300s, 600s and 900s. Then, a constant shear rate of $\dot{\gamma} = 1s^{-1}$ was applied and rheological measurements were made each 20s during 400s. For each resting time a new grout sample was prepared.

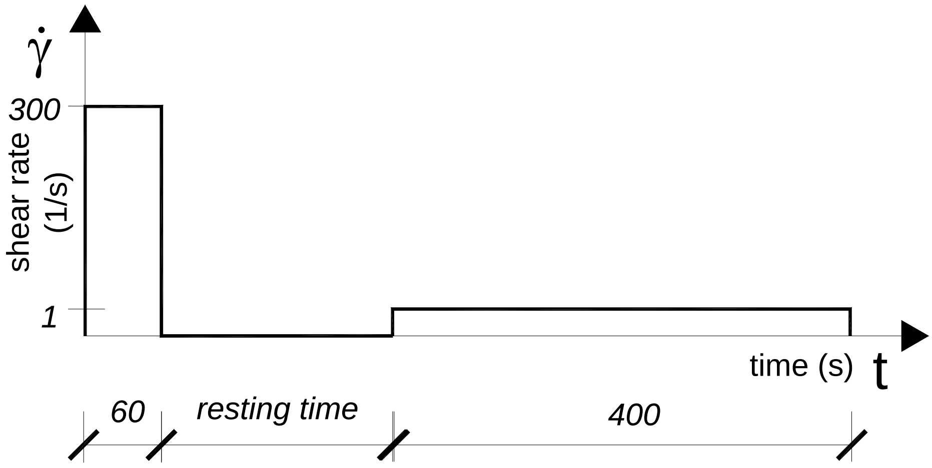

Figure 6.2: Procedure 1 for thixotropy test.

The expected steady state apparent viscosity at this shear rate was calculated from the rheological parameters measured. This procedure also enables the determination if the imposed deformation is able to break the state of flocculation built at rest and if there is any irreversible evolution of the grout tested.

6.4.3.2 *Procedure 2*

Procedure 2 (Fig. 6.3): After the pre-shear, different shear rates were applied ($\dot{\gamma} = 5s^{-1}, 10s^{-1}, 50s^{-1}, 100s^{-1}, 300s^{-1}, 500s^{-1}, 1000s^{-1}$) to the sample. Each shear rate was applied 60s after the resting time and during 600s. This enables the determination of the maximum shear rate that could be applied to the sample without destroying the microstructure. High shear rate will generate a different structure in the hydraulic binder based particle suspension and will disperse excessively the particles. Non homogeneity of grout will be obtained.

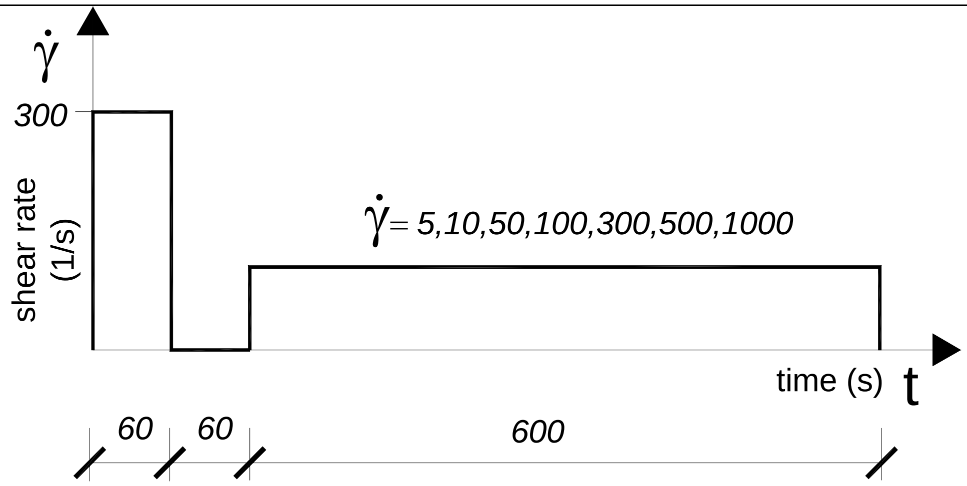

Figure 6.3: Procedure 2 for thixotropy test.

6.5. Results and discussion

6.5.1 Rheological measurements- *time independent properties and temperature effect*

The procedure applied to NHL5 and to NHL5+15% fly ash based grouts enabled the determination of flocculation and de-flocculation area for each temperature test, due to step up and down procedure. During grout injection inside a porous media like masonry the flow tends to stop enabling an increase of the particle flocculation phenomenon. Fig. 6.4 and 6.5 present flocculation areas according to the procedure adopted.

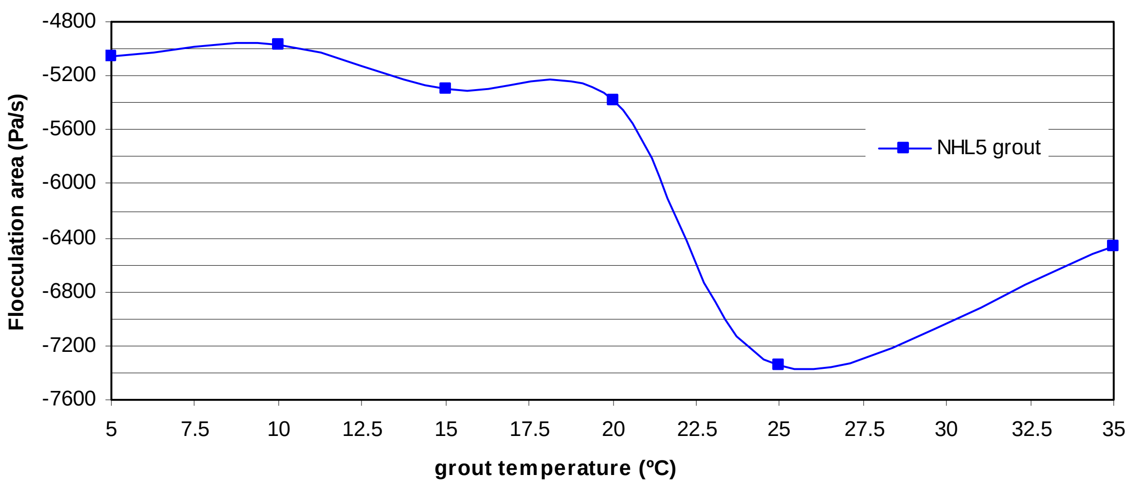

Figure 6.4: Flocculation area of NHL5 based grout according to the procedure adopted.

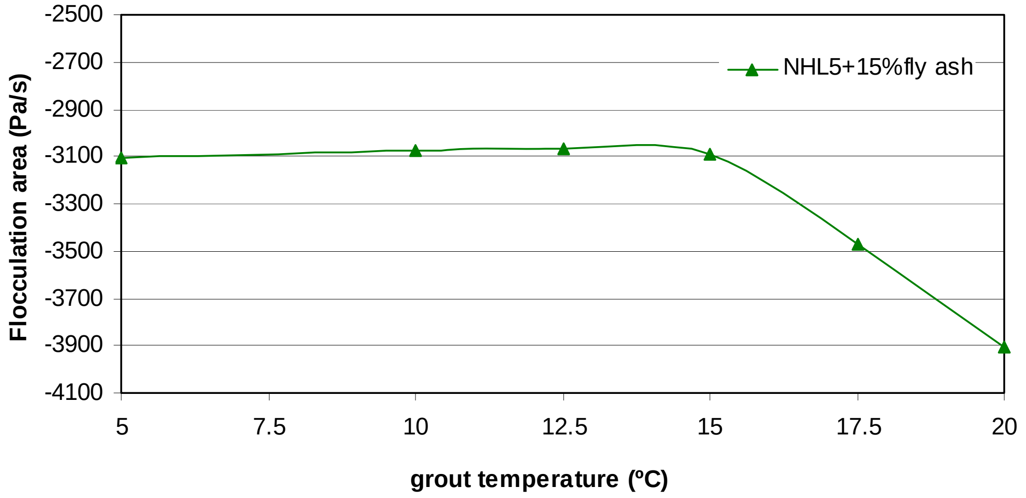

Figure 6.5: Flocculation area of NHL5+ 15%fly ash based grout according to the procedure adopted.

According to the previous results it seems that there is a grout threshold temperature (T_{limit}) that separates a domain where flocculation area is almost constant, from another where flocculation area starts significantly to increase. Probably, this means that in the first region thixotropic effects are almost isolated from the irreversible effects (due to hydration). The same does not happen in the second region, where a temperature increase leads to faster hydration reactions. Fig. 6.6 and 6.7 present the de-flocculation area for the same tested grouts.

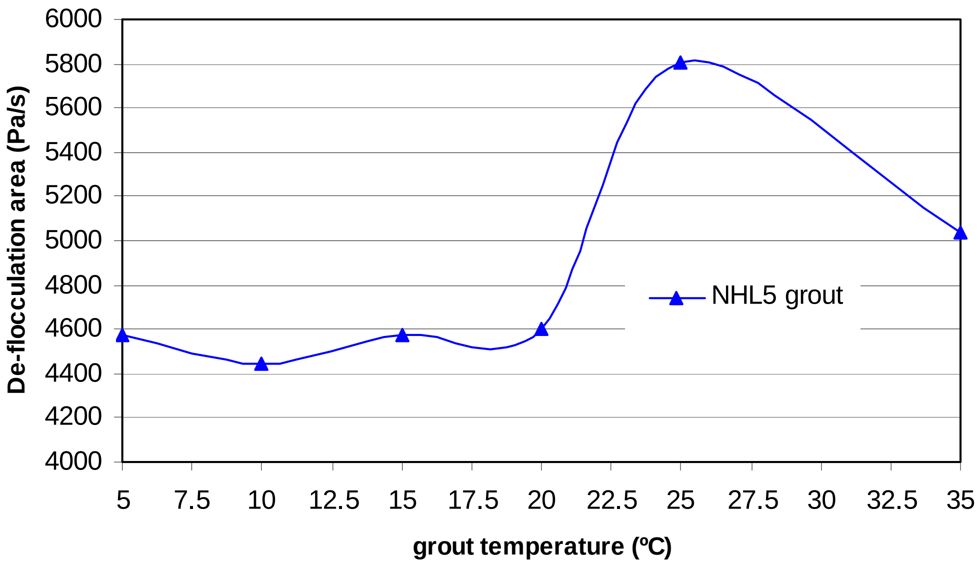

Figure 6.6: De-flocculation area of NHL5 based grout according to the procedure adopted.

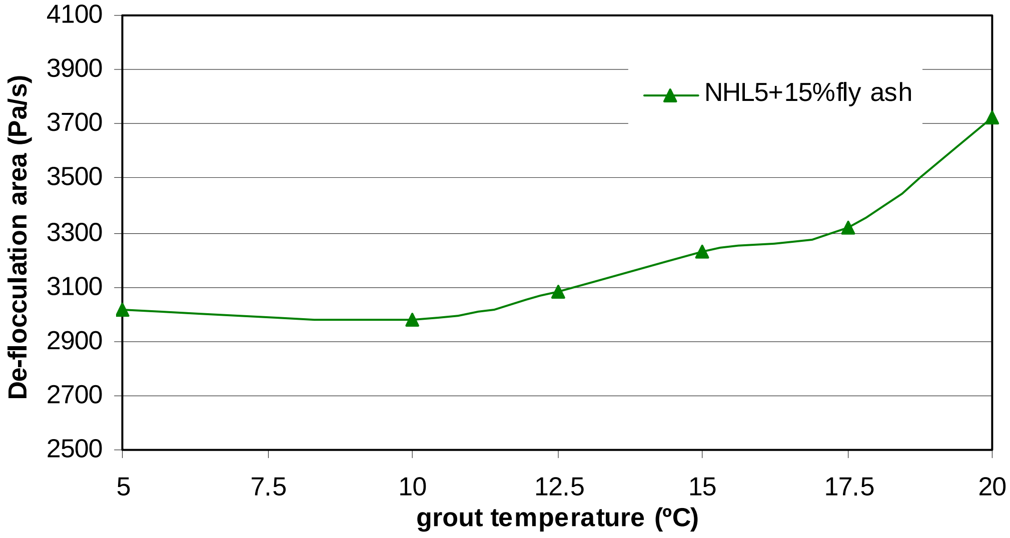

Figure 6.7: De-flocculation area of NHL5+ 15%fly ash based grout according to the procedure adopted.

Table 6.3 present the results of the T_{limit}, flocculation and de-flocculation values for the equilibrium situation (the first region).

Table 6.3: Results of the T_{limit}, flocculation and de-flocculation values for the equilibrium situation (the first region).

	T_{limit}	$Flocculation_{equilibrium}$	$De\text{-}Flocculation_{equilibrium}$
NHL5 based grout	20 ºC	-5200 Pa/s	4500 Pa/s
NHL5+15%fly ash based grout	15 ºC	-3100 Pa/s	3000 Pa/s

Like in the measurement of thixotropy in a loop test, the flocculation and de-flocculation areas also have the dimension of energy related to the volume of the sample sheared (Eqn.6.3).

$$A = Pa \cdot s^{-1} = \frac{N.m}{s} . \frac{1}{m^3} = \frac{energy}{volume} \tag{6.3}$$

For flocculation phenomenon, the area A of the flow curve in the range $300s^{-1}$ - $0s^{-1}$ (step down test) indicates the energy that is required to build-up the thixotropic structure. In the other hand, de-flocculation values are related to the energy that is required to break down the thixotropic structure. This energy can not be characterised as a material parameter in the same way as the Bingham parameters (for example) as the area A depends on the shear history of the material and degree of dispersion. However, the same experimental procedure was adopted to ensure the same grout initial conditions in order to let results comparison.

Comparing the flocculation and de-flocculation area values of the two analysed grout samples, it was detected that flocculation area values are higher than the de-flocculation ones. Thus, higher energy is necessary to build-up the microstructure than to break it down.

The time for structural breakdown is normally considerably shorter than the time for the structural build-up due to the high shear rates applied during the breakdown. During the structural build-up, diffusion and thermal motion control the process which leads to a long build-up time since they lead to very small effects compared to the shear rate (Barnes *et al.,* 2001), (Barnes, 1997), (Billberg, 2006). Since the time for build up was the same for breakdown in the tested procedure, the previous sentence probably may explain the results (Table 6.3) because it means that the time scale to get the same microstructure in the flocculation situation should be higher than for the de-flocculation.

It was also detected that flocculation and de-flocculation values are higher for NHL5 based grouts, meaning that it is more thixotropic than NHL5+15% fly ash based grout. Probably this happens because in the first grout there are more elongated particles than in the one that contain fly ash (spherical shape). In fact, Barnes (Barnes, 1997) refers that thixotropy is more pronounced in systems containing non-spherical particles because they have to find themselves in the best 3D structure by rotation, as well as movement, and progress from a completely structured situation to a completely unstructured one, giving a shear thinning response.

After the determination of T_{limit} it was decided to adopt the minimum found simultaneously in NHL5 and in NHL5+15% fly ash based grouts. So, T_{limit} =15ºC was adopted in the following procedures.

The Sisko model was chosen for the determination of the steady state parameters. The modified Bingham equation was chosen for yield stress and plastic viscosity determination. Table 6.4 presents the values of those rheological parameters.

Table 6.4: Values of viscosity in the second Newtonian region (η_∞), consistency (K_2), power-law index (*n*), yield stress (τ_0) and plastic viscosity (η_p) for NHL5 based grouts and NHL5+15% based grouts tested.

	NHL5 fly ash=15% w/b=0.70	**NHL5 w/b=0.70**
η_{00}	0.05 Pa.s	0.08 Pa.s
K_2	0.29 $Pa.s^n$	0.68 $Pa.s^n$
n	0.55	0.40
τ_o	0.42 Pa	1.04 Pa
η_p	0.10 Pa.s	0.15 Pa.s

6.5.2 Rheological measurements- time dependent properties

6.5.2.1 *Procedure 1*

The expected steady state apparent viscosity at $\dot{\gamma} = 1s^{-1}$ was calculated from the rheological parameters measured according to procedure 1. Assuming that Sisko model is enough for a steady state characterization, then:

$$\eta = \eta_\infty + K_2 \dot{\gamma}^{n-1} \tag{6.4}$$

So, for $\dot{\gamma} = 1s^{-1}$

$$\eta_{st}(1) = \eta_\infty + K_2.1 \tag{6.5}$$

Eqn. 6.10 could also be written in the following way:

$$\tau = \tau_\infty + K_2 \dot{\gamma}^n \tag{6.6}$$

Knowing that there is a linear increase of yield stress with time (linearity was experimentally confirmed), causing the yield stress to be called the apparent yield stress (Roussel *et al.*, 2007), then:

$$\tau_\infty = \tau_0 (1 + \lambda) \tag{6.7}$$

and according to (Roussel, 2006), the structural parameter (λ) that expresses the time evolution of the microstructure from a non-equilibrium state to an equilibrium one could be written in the following way:

$$\frac{d\lambda}{dt} = \frac{1}{T} - \alpha\lambda\dot{\gamma} \tag{6.8}$$

where the first term on the right hand side of (Eqn. 6.8) refers to the flocculation state and the second to the breakdown of the structure (T and α are thixotropy parameters).

$$\text{At rest} \begin{cases} \dot{\gamma} = 0 \\ \alpha\lambda\dot{\gamma} = 0 \end{cases}$$

$$\lambda = \int \frac{1}{T} dt \Leftrightarrow \lambda = \frac{t}{T} \tag{6.9}$$

$$\Rightarrow \tau_{\infty}(t) = \tau_0 + \tau_0 \frac{t}{T} = \tau_0 + A_{thix} t \tag{6.10}$$

where A_{thix} is the rate of flocculation in Pa/s.

Just after mixing, the grout structural parameter is zero $(\lambda = 0)$. After grout injection, the resting time becomes higher and λ will evolve from its initial zero value to a positive one, depending on the thixotropy of each material. The combination of Eqn. 6.7 and 6.10 leads to:

$$\tau = \tau_0 + A_{thix} t + K_2 \dot{\gamma}^n \tag{6.11}$$

For the procedure applied in this situation it was considered $\dot{\gamma} = 1s^{-1}$, so, the first derivate of

$$\tau = \tau_0 + A_{thix} t + K_2 \tag{6.12}$$

will give the flocculation (or structuration) rate of each grout tested.

The viscosity obtained with the procedure 1 for the two types of grout tested is presented in Fig. 6.8 and 6.9.

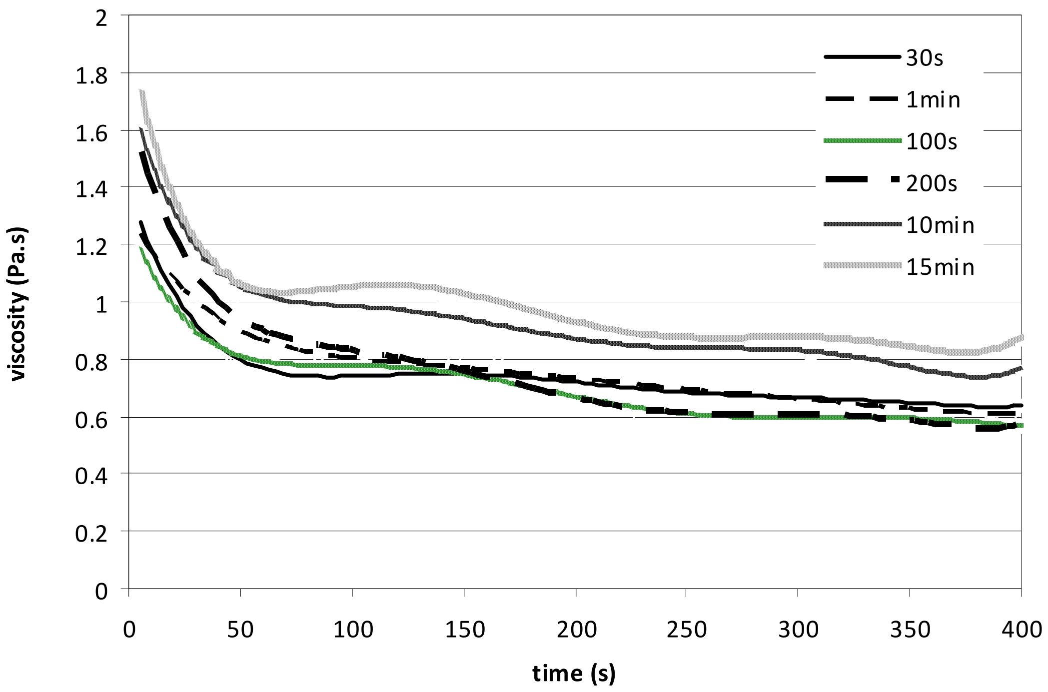

Figure 6.8: Viscosity of NHL5 based grouts according to the procedure 1.

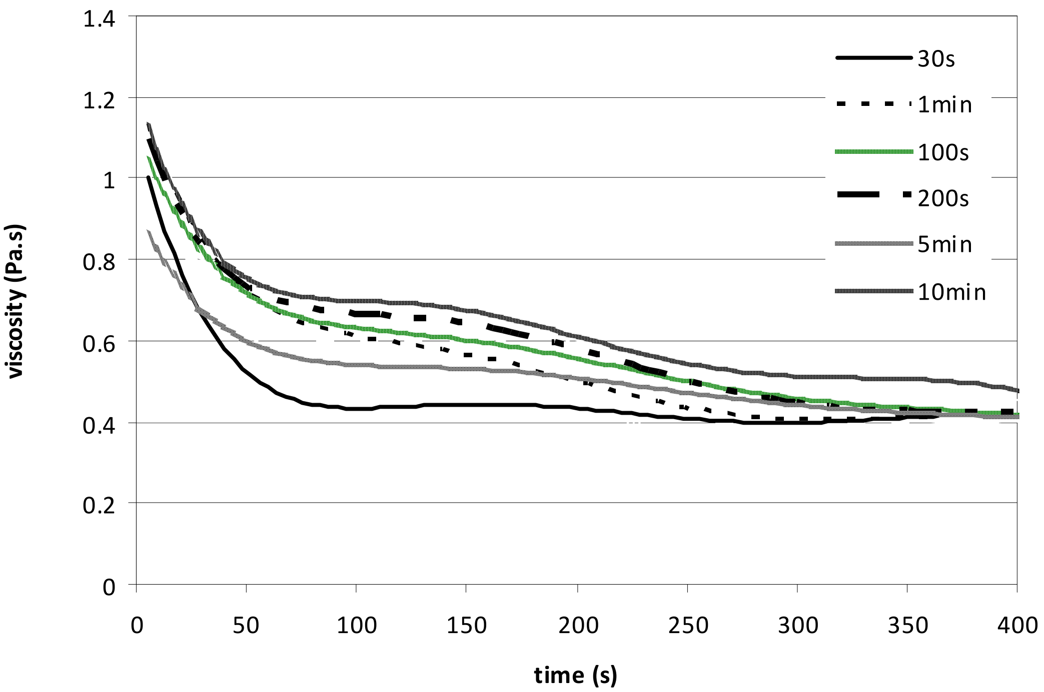

Figure 6.9: Viscosity NHL5+15%fly ash based grouts according to the procedure 1.

The expect steady state viscosity, given by Eqn. 6.5 taking into account the values in Table 6.4, for NHL5 based grout is $\eta_{st}(1) = 0.08 + 0.68 = 0.76 Pa.s$ and for NHL5+15%fly ash the value is $0.34 Pa.s$. A comparison between the experimental values (constant values for viscosity in Fig. 6.8 and 6.9) and the steady state viscosity show a good approximation between those two values for almost resting time studied here. The best correlation is for a resting time in the range between 30s and 60s. However, for a resting time higher than 10 minutes the differences are too high, meaning that the deformation imposed with the rheometer is not able to break the state of flocculation built at rest and probably irreversible evolution of the grout tested is under development.

For each resting time tested (until 10 min) the initial shear stress obtained at a very low shear rate (trying to simulate the behaviour at rest) was recorded. For that, $\dot{\gamma} = 1s^{-1}$ was adopted since lower shear rates lead to inaccurate measurements. Fig. 6.10 and 6.11 present those results.

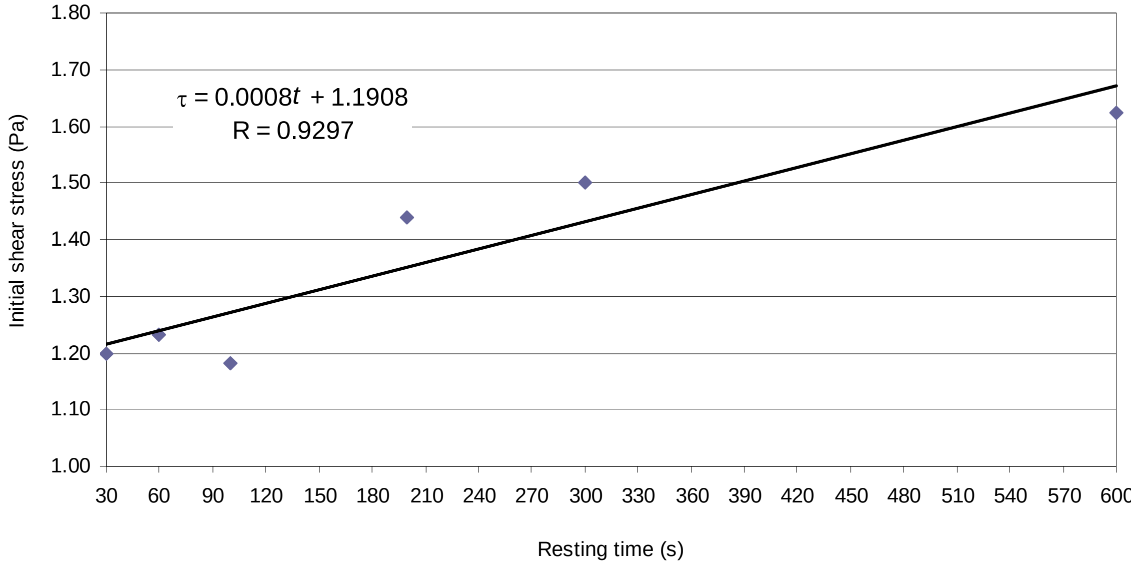

Figure 6.10: Initial shear stress obtained at a very low shear rate for NHL5 based grout after resting time.

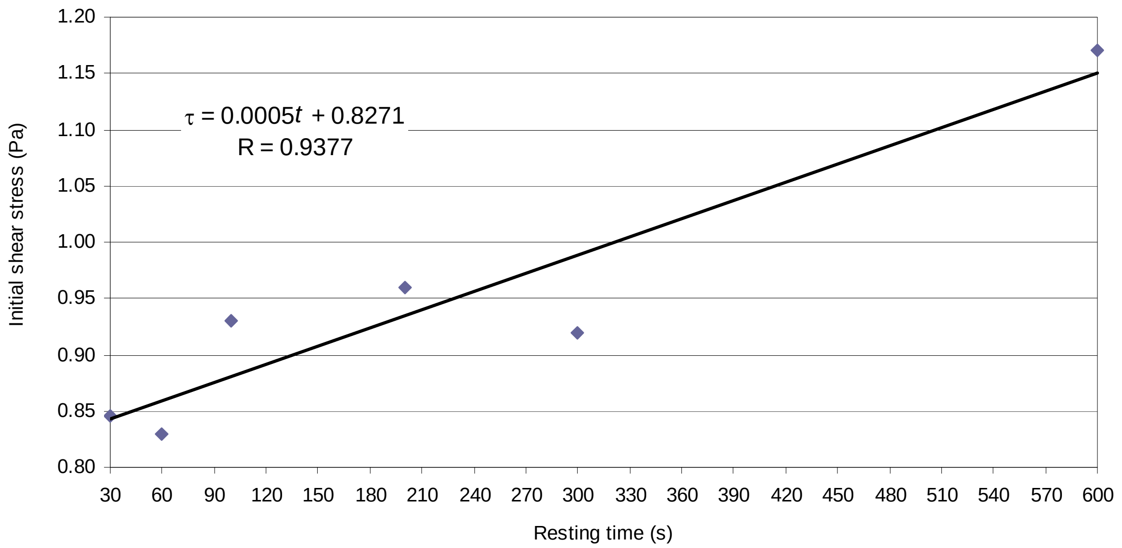

Figure 6.11: Initial shear stress obtained at a very low shear rate for NHL5+ 15% fly ash based grout after resting time.

According to Eqn. 6.12, A_{thix} should be approximately the slope of the functions represented at Fig. 6.10 and 6.11. So, $A_{thix} = 0.0008 Pa/s$ for NHL5 based grouts and $A_{thix} = 0.0005 Pa/s$ for NHL5+15%flys ash based grouts. This means that the structuration rate of NHL5 is almost double of NHL5+15% fly ash. The slope is the rate of increase of the apparent yield stress of the material at rest and may be used in thixotropic evaluations.
However, those values of structuration rate obtained for these grouts are really low comparing to other values mentioned in bibliography of SCC, where thixotropic concretes present a A_{thix} at least between 0.1 and 2 Pa/s (Roussel, 2006), (Billberg, 2006).

The thixotropic behaviour of cement grouts is related to flocculation and de-flocculation, as it occurs in NHL5 grouts. The Hattori and Izumi theory- the Coagulation Rate theory- describes these phenomena in particle suspensions and with that the apparent viscosity of the suspension could be calculated as function of time. The theory is related to the viscosity of the suspension, friction coefficient between particles, number of particles, initial degree of dispersion, coagulation rate constant and time. The term coagulation describes the occurrence when two or more cement particles come into contact with each other for a certain time. That phenomenon is a result of the total potential energy interaction that exists between particles.

The finer the cement is, the more the colloidal forces will influence its movement, and thereby the stronger will the coagulation process be (higher thixotropic effect). Among that, the finer the cement is, the more the number of particles will increase as well as the specific surface. The connections between the particles formed by the total potential energy interaction could be reversible junctions or/and permanent junctions. According to J. Wallevik (Wallevik J., 2009) the particles that are influenced by the total potential energy effects are considered to be colloidal particles. They are the ones within $1nm$ to $1\mu m$. However, he demonstrated that cement particles $\leq 40\mu m$ in diameter seem to be able to behave as colloidal particles and this means that coagulation will occur easily.

Comparing NHL5 particles dimensions (75% of particles present a dimension $\leq 90\mu m$) (see Table 6.1) with cement particles dimensions (90% of cement particles present a dimension $\leq 90\mu m$, according to tests made in our lab), it could be found a possible explanation for the smaller flocculation rate of natural hydraulic lime grouts.

6.5.2.2 *Procedure 2*

In order to understand what is the behaviour of grouts under different shear rates procedure 2 was applied. The goal was the determination of the maximum shear rate that could be applied to the sample without destroying the microstructure.

For different rate of shear applied to grouts viscosity and shear stress values were recorded. Fig. 6.12, 6.13 present the relationship between viscosity and time, at a constant resting time of 60s, for the two tested grouts.

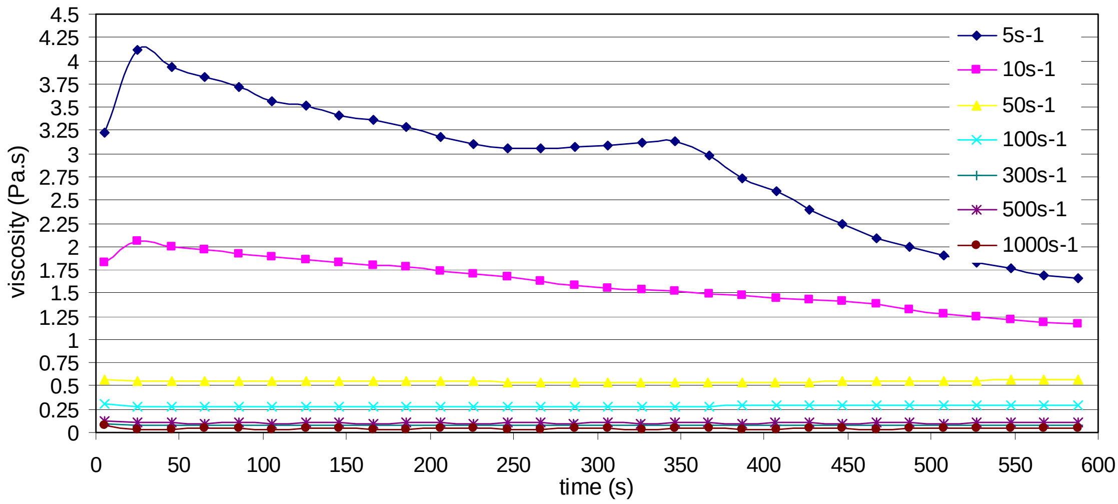

Figure 6.12: Viscosity of NHL5 based grouts according to the procedure 2.

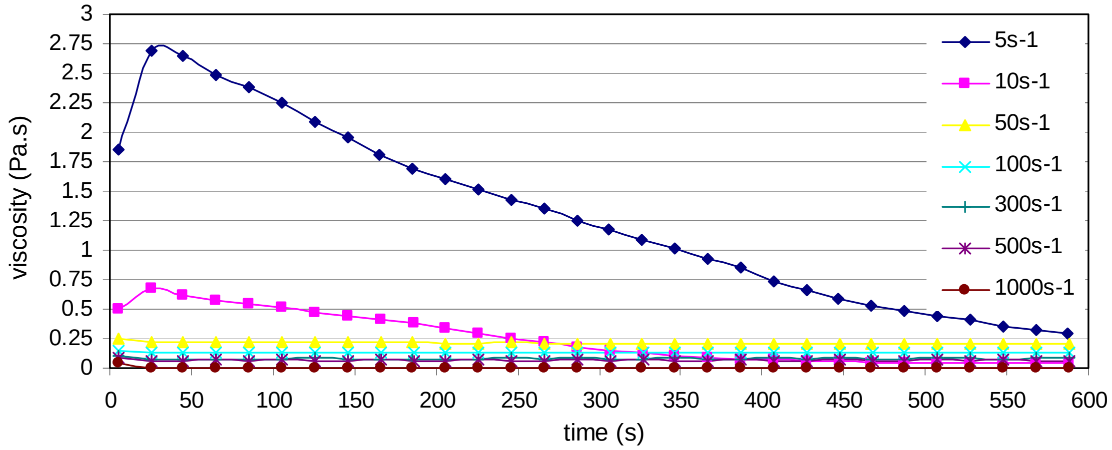

Figure 6.13: Viscosity of NHL5+15%fly ash based grouts according to the procedure 2.

The previous figures show that for $\dot{\gamma} \geq 10s^{-1}$ 300s are enough to get the steady state. For $\dot{\gamma} > 50s^{-1}$ both grouts reach fast the steady state which means that no significant improvement will occur for higher rates of shear. Probably the determination of this range enables the control of viscosity in the sense that for $\dot{\gamma} > 50s^{-1}$ this value is constant.

It was made an experimental evaluation of the shear rate that the lab mixer applies. The grouts are mixed at a shear rate in the range of magnitude between $10s^{-1}$ and $100s^{-1}$ which means that the previous results will help in grout rheological characterization, since $50s^{-1}$ fits in the previous range. In fact, Barnes in its state of art concerning thixotropy (Barnes, 1997) mentioned the easy way to characterise the flow of thixotropic materials in mixes if it is assumed that the mixer behaves in the same way as a rheometer, running at the same shear rate as the average shear rate in the mixer. The important issue is to equal shear rate mixer to the shear rate of rheometer in order to get a good rheological characterization.

The relationship between shear stress and time (at a constant resting time of 60s) for the two tested grouts is present in Fig. 6.14 and 6.15.

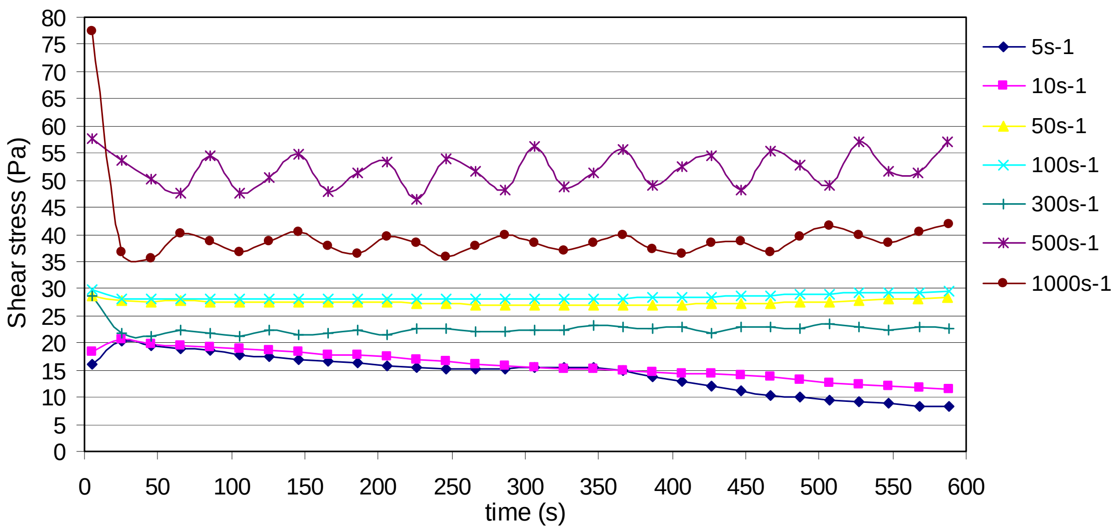

Figure 6.14: Shear stress of NHL5 based grouts according to the procedure 2.

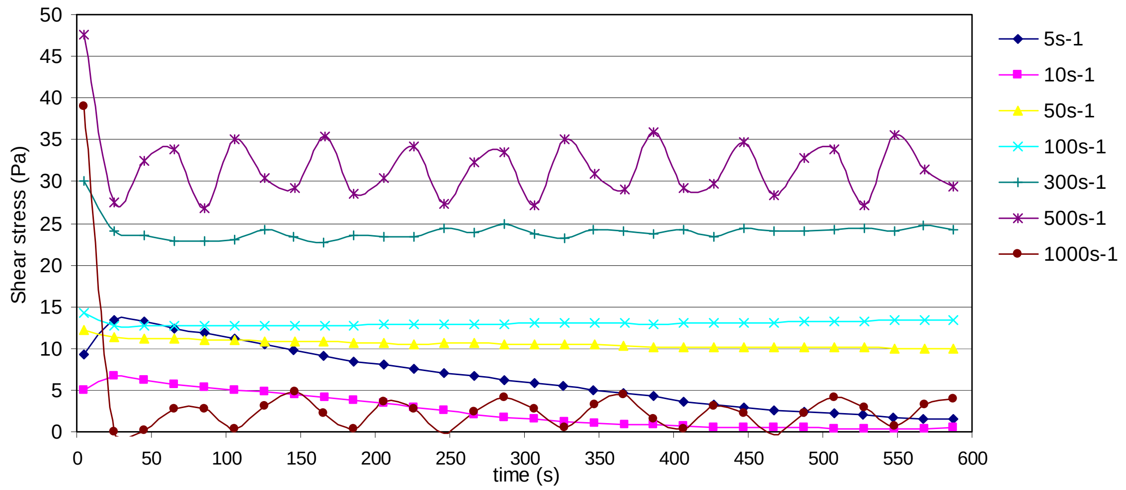

Figure 6.15: Shear stress of NHL5+15% fly ash based grouts according to the procedure 2.

Here it could be said that the maximum shear rate that could be applied to the sample without destroying the microstructure is less than $500s^{-1}$ for both grouts. The inaccurate results obtained may be explained by the fact that high shear rate will generate a different structure in the hydraulic binder based particle suspension and will disperse excessively the particles. Non homogeneity of grout will be obtained. This determination also supplies information about the maximum pre-shear that may be used in tests and also during grout mixing procedure.

The analysis of Fig. 6.14 and 6.15 lead to the conclusion that the shear stress of the two tested grouts for a shear rate between 50 and $100s^{-1}$ is almost constant. However, shear stress of NHL5 based grout is higher than for grout with fly ash (27Pa and 12Pa respectively). A detailed analysis of the breakdown structure represented by the area between the initial shear rate and the steady shear rate applied to the grouts tested (Fig. 6.16) proves that. The blue bars represent NHL5 grouts and the green ones represent NHL5+15% fly ash based grouts. Filled bars mean initial shear stress and bars with diagonal pattern represent equilibrium shear stress.

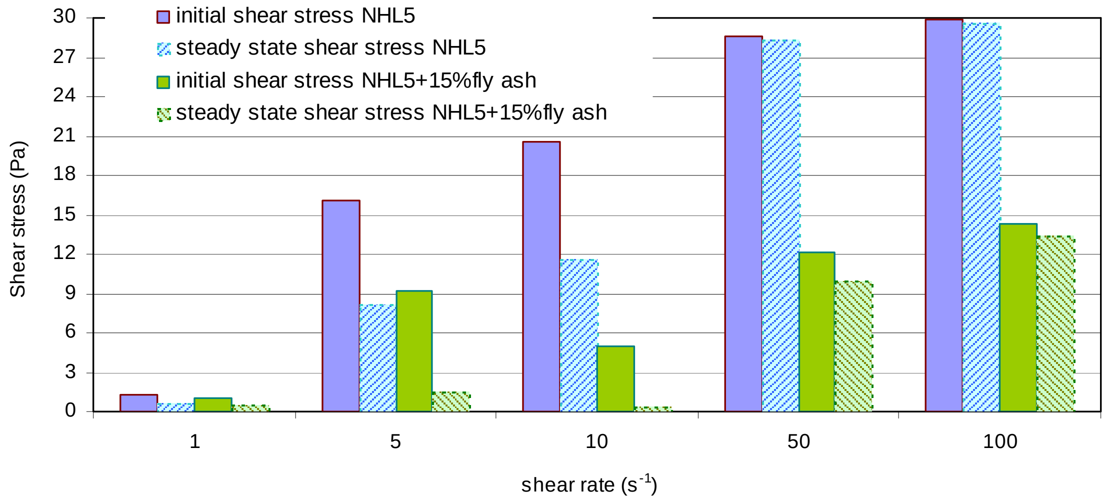

Figure 6.16: Initial shear stress (filled bars) and corresponding steady state shear stress (diagonal pattern) for NHL5 based grout (blue colour) and NHL5+15%fly ash based grouts (green colour).

The result show that if a part of natural hydraulic lime (NHL5) is replaced by fly ash, the thixotropy decrease. Our previous results also show that yield stress and plastic viscosity decrease with fly ash addition. A reduction of the yield stress value was also detected in others research works when cement was partially replaced by pulverised fly ash in cement based grouts (Sonebi, 2006). Fly ash improves the contact between the particles of cement by ball bearing effect and reduces the friction forces, therefore reducing the applied shear stress needed to initiate flow (less yield value), which may explain the smallest initial shear stress values for NHL5 grout with fly ash in Fig. 6.16.

6.6 Main conclusions

A research work was developed which aims at contributing to better understand the flow behaviour that natural hydraulic lime-fly ash grouts presents under different temperatures. Two grout compositions were tested: hydraulic lime grout with water/binder (w/b) content equal to 70% and hydraulic lime with 15% of fly ash with w/b=70%. For that purpose, the measurement programmes designed in this work aims at establishing a determination of the thixotropy of each grout and thus compare how structured are the mixes in relation to their variables of preparation and test conditions.

An attempt to find the temperature limit that isolates thixotropy effect from hydration reactions in a grout was made. Maximum resisting time, rate of flocculation and analysis of the hydraulic lime grout behaviour tested at different shear rates were performed concerning certain rheological models and hypothesis.

According to the previous results (Fig. 6.4 and 6.5) it seems that there is a grout threshold temperature (T_{limit}) that separate a domain where flocculation area is almost constant, from another where flocculation area starts significantly to increase. Probably, this means that in the first region thixotropic effects are almost isolated from the irreversible effects (due to hydration). The same does not happen in the second region, where a temperature increase leads to faster hydration reactions. For NHL5 based grout T_{limit} =20ºC and for NHL5+15%fly ash based grout T_{limit} =15ºC.

It is possible to estimate the expected viscosity value of steady state for the tested grouts taking into account the Sisko model, concerning that the maximum grout resting time is not higher than 10min.

An effort to take into account microstructure, shear and time dependence was made and a flocculation rate for each grout tested was calculated due to its very usefulness in grout characterization and design for masonry injection purpose.

For a shear rate higher than $50s^{-1}$ it was shown that viscosity values are constant for both grouts during the 600s of test (procedure 2). Since grout mixing also occurs in the same shear rate magnitude ($10\text{-}100s^{-1}$) and mixing time is not higher than 360s it means that it is really possible to characterize the flow of those thixotropic materials in mixes (assuming that the mixer behave in the same way as a rheometer). For $\dot{\gamma} \geq 10s^{-1}$ 300s are enough to get the steady state in those grouts.

The maximum shear rate that could be applied to the sample without destroying the microstructure is less than $500s^{-1}$ for both grouts. This determination also supplies information about the maximum pre-shear that may be used in tests or that could be used for grout mixture on field. $300s^{-1}$ seems to be a good option.

Addition of 15% of fly ash reduces NHL5 grout yield stress and plastic viscosity, as well as thixotropy.

6.7 References

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Chapter 7. Grouts for masonry injection: how to select injection parameters - *first approach*

7.1 Purpose

Beyond the study of the best fresh grout properties (like water retention capacity, stability, rheological properties, flow time, among others) and hardened properties (such as mechanical strength, shrinkage, porosity, etc), the study of the interaction between that optimized grout and masonry is of major importance.

The lack of information about which is the best solution for grout design, as a function of the porous media to be injected, enhances the importance of a detailed research on the subject. Thus, to improve the knowledge about the physical mechanisms that take place during injection and that determine the penetration of the grout in the masonry, reproducible masonry samples of cylindrical shape were used. Injection tests were performed in those cylindrical samples. The study has envisaged to define an adequate composition for grouts to be used for the strengthening of walls. It was shown that rheological properties are of major importance and have a great influence in grout design and injectability, as confirmed by the injection tests. The Marsh cone method (a flowability test used for specification and quality control of grouts) was also used in tests and seems to be inadequate for grout design for injection purpose at least if the masonry grain size distribution is equal or smaller than the minimum grain size distribution tested. The test method adopted also enabled a grout design regarding several factors like the best injection pressure to adopt and the best w/b ratio, among others.

The results were presented in (Bras *et al.,* 2010). An attempt to get an injectability curve for each grout tested as a function of % of total mass that passes through the fine grained soil was also made. After 45 and 120 days, the injected cylinders were subjected to the splitting test in order to define the tensile strength of the "repaired" material.

7.2 Introduction

In practice, the variability of void structures within masonry requires an ability to fine tune the rheological properties of a grout, in order to optimally fill a given void space. Hence the grout specification involves knowledge of the flow capacity within the porous media and compatibility with the original materials used in masonries. Grout flow properties are affected by a large number of parameters (Bras *et al.*, 2009), (Fernàndez-Altable *et al.*, 2006), (Eriksson *et al.*, 2004).

A suitable grout composition, together with the flow capacity and the ability to fill the voids, will regulate the consolidation quality (Toumbakari, 2002), (Van Rickstal, 2000), (Binda *et al.*, 2003), (Binda *et al.,* 2003b), (Baronio *et al.*, 2003). Hence, and as a first step, the ability of a grout to perform as desired is commonly assessed using flow properties measured in laboratory, such as its yield stress and plastic viscosity, among others. It is clear, however, that additional characterization of the flow and injection processes are needed, due to the complex composition of grouts and the interaction of flows and flow geometries which may evolve in time as a result of phase segregation and deposition for example.

Injection grouts based on hydraulic lime, limestone or cement, are colloidal suspensions in which the particle interactions may lead to the formation of various microstructures. Depending on how such structures respond to an applied shear stress or strain rate, one observes different types of macroscopic flow behaviour (Mewis and Spaull (Mewis *et al.*, 1976), Coussot and Ancey (Coussot *et al.*, 1999), Bras and Henriques (Bras *et al.*, 2009). These rheological properties may be evaluated in bulk through a rotating viscometer. In this type of device, a rotating element is placed into a grout specimen and is then rotated at a certain velocity by a motor. The force caused by this rotation (torque) is proportional to the viscosity and a shear stress (τ) is generated inside the grout. The angular velocity of the rotating element gives rise to a velocity gradient in the grout, the shear rate ($\dot{\gamma}$). The ratio between shear stress and shear rate is called the apparent viscosity of the fluid (η).

The grout specification should be optimized for injection purposes, which means that even for simple binder and water grout compositions their properties especially in the fresh state, like rheological and stability properties, should be improved (Toumbakari, 2002), (Van Rickstal, 2000), (Bras *et al.,* 2008), (Bras *et al.*, 2009).

On field, the rheology of grouts is often characterized by its workability. This property of a grout is a performance index representing how easily it is injected. There are many test methods to evaluate the workability. One of them is called the Marsh cone test (a flowability test) used for specification and quality control of grouts, according to standards such as (EN 445, 2000) or (ASTM C939-02). The measured flow time according to these standards is connected to the grout fluidity. It means that the longer the flow time, the lower is the fluidity. International bibliography refers several studies concerning the relation between flow time and rheological parameters (like) for cement grouts (Roussel *et al.*, 2005), (Nguyen *et al.*, 2006). However, the determinations of yield stress and plastic viscosity are performed without considering the interaction between grout and masonry (the porous media to be injected).

The penetration of grouts is determined by the interaction between grout and masonry. It means that beyond the study of the best fresh grout properties (like water retention capacity, stability, rheological properties, flow time, among others) and hardened properties (such as mechanical strength, shrinkage, etc), the study of the interaction between that optimized grout and masonry is of major importance. The internal structure of the masonry, the permeability, the pore size distribution and the moisture content are the most important properties of the masonry as far as injectability is concerned (Toumbakari, 2002), (Van Rickstal, 2000), (Binda *et al.*, 2003).

The lack of information about which is the best solution for grout design, as a function of the porous media to be injected, enhances the importance of a detailed research on the subject. Thus, to improve the knowledge about the physical mechanisms that take place during injection and that determine the penetration of the grout in the masonry, reproducible masonry samples of cylindrical shape were used.

Chapter 7 analyses some relevant grout properties such as yield stress, viscosity, bleeding, flowability and injectability capacity for natural hydraulic lime based grouts (EN459-1 NHL5). It shows that there are substantial variations on grout properties as a consequence of different water/binder ratios (w/b) and tries to increase the understanding of the influence of the mixing design in grouts behaviour and how it may improve some essential injection characteristics. For that purpose, injection tests in cylindrical samples trying to reproduce masonry materials were made. An attempt was made to achieve an optimal solution for grout design and to get an injectability curve for each grout tested as a function of % of total mass that passes through the fine grained soil was also made.

When a good filling and a good bonding of the grout to the masonry original material are achieved, the load bearing capacity of the structure will significantly improve after curing of the grout. Thus, the importance of an optimal grout composition from a fresh and an hardened state is crucial. In order to study its bond strength, it was adopted the splitting test of the previous injected cylinders.

7.3 Material Studied

7.3.1 Field of use and materials selection

In principle the selection of a binder to be used in grouts for injection should take into account the compatibility with the original material to be injected. Therefore, the present research program used only the grouts made according to Chapters 4, 5 and 6.

In order to address the influence of different water content used in the production of grouts, a simple mix consisting of NHL5 natural hydraulic lime (according to EN459-1) and water has been considered. The water/ binder ratios (w/b) tested were the following: 55%, 60%, 70% and 80% in weight. The mixing procedure adopted to produce grouts was the best solution according to (Bras *et al.*, 2009).

Injectability tests were made in order to study the penetrability of the grouts. Since it is hard to reproduce a real masonry and because it is difficult to visualize what is happening inside the porous media being injected by grout, masonry samples were created using a certain type of sand. So, to improve the knowledge about the physical mechanisms that take place during injection and that determine the penetration of the grout in the masonry, transparent Plexiglas cylinders, diameter 144 mm and height 300 mm, are filled with a fraction of crushed limestone sand and are injected unidirectionally from bottom to top. These types of aggregates were adopted since they have, just like masonry, a water absorbing action. The sand were bought from a quarry company, washed out, dried and sieved to obtain different grain size distributions to enable the simulation of different permeability for the masonry. Two different grain size types were adopted: type “2-4”mm and “4-10”mm.

7.3.2 Material characteristics

The hydraulic lime used is a EN459-1 NHL5 produced in Portugal by Secil-Martingança, which has the characteristics presented in Table 7.1 according with the information of the quality control system provided by the manufacturer. Expansibility test for hydraulic lime was made according to EN 196-3.

The grain size distribution as well binders' fineness (NHL5 and fly ash) is represented in Chapter 6.

Table 7.1: Natural hydraulic lime characteristics.

Compression resistance at 7 days (MPa)	**5,5**	
Setting time	start	**2h45**
	end	**6h37**
Expansibility	**0,79mm**	
Free lime	**3,89%**	
SO_3	**3.23%**	
Ignition loss	**19.84%**	

Table 7.2 presents chemical characterizations of NHL5 according to scanning electron microscope (SEM) results.

Table 7.2: Chemical characterizations of NHL5 according to SEM results.

Formula	**(%)**
Na2O	0.96
MgO	0.78
Al2O3	1.43
SiO2	62.88
P2O5	1.01
K2O	0.52
CaO	5.54
TiO2	3.3
MnO	9.75
Fe2O3	12.54
ZrO2	1.29

Fig. 7.1 represents the first grain size distribution (type "2-4"mm) of the limestone sand used in injection tests. With this grain size distribution the authors want to simulate voids with 2.7mm, which correspond to d_{50} (the diameter through which 50% of the total sand mass is passing) inside the porous media.

A coarser sand was also used (fraction type "4-10"mm) and the same analysis was made. A comparison between the two types of porous media ("2-4"mm and "4-10"mm) was made for the injection results.

Injectability tests were also made in several porous media in order to study the penetrability of the grouts. Masonry samples were simulated by using five different crushed limestone sands: "0-4"mm; "0-2"mm; "0-10"mm; "2-4"mm; "4-10"mm (see Fig. 7.2).

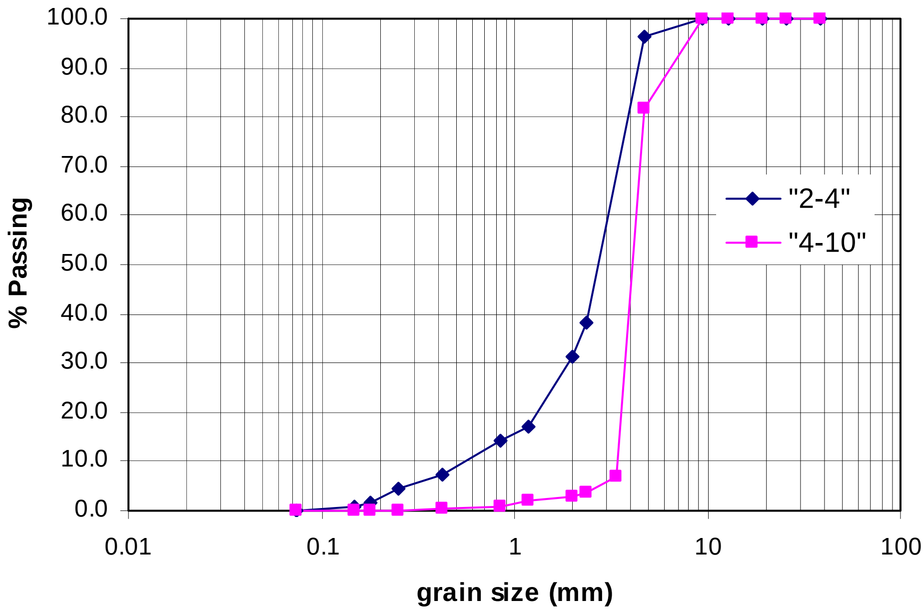

Figure 7.1: Grain size distribution for sand type "2-4" mm and "4-10"mm used for cylinders grout injection.

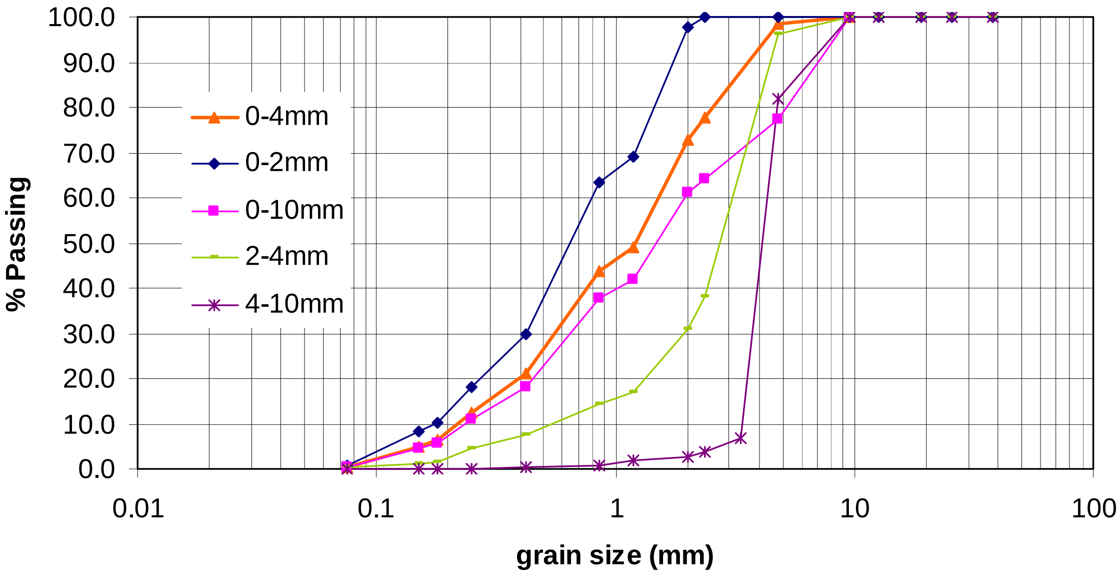

Figure 7.2: Grain size distribution for sand types “0-4”mm; “0-2”mm; “0-10”mm; “2-4”mm and “4-10”mm used for cylinders grout injection.

7.4 Procedure

7.4.1 Mixing procedures

Before preparing the grouts the dry hydraulic lime was hand mixed with a trowel to avoid the formation of granules. The mixer blade had a helicoidal shape, with a diameter a little smaller than the cup diameter in order to allow all the grout to be mixed. The gap at the bottom, between the blade and the cup was 4mm. Ordinary tap water was used for the preparation of the grouts. The water was allowed to flow freely until a stable water temperature of 18ºC was reached.

Several mixing procedures were tested in order to understand their influence in natural hydraulic lime grout behaviour (Bras *et al.*, 2009). The adopted mixing procedure is referred in Chapter 6. The w/b ratios (in weight) studied were the following: 0.55, 0.60, 0.70 and 0.80. For each test, three samples were taken for every mixing procedure. The relative humidity of the laboratory was 55% and the temperature 20±2 ºC.

7.4.2 Study of grout bleeding

The bleeding test used in this research was based on ASTM C 940. According to this standard 800 ml of freshly mixed grout was poured into a 1000 ml glass graduated cylinder and covered. The height of bleed water was noted after complete sedimentation. The final bleeding was calculated according to the expression (ASTM C940-98a, 2003). The main purpose of the test was to evaluate the influence of mixing procedures on the stability of the grouts. An excess of bleeding in a grout means that there is a substantial amount of free water on the surface of the unset grout.

7.4.3 Marsh cone test

The Marsh cone test was based on EN445 (EN 445, 2000). The measured flow time (FT) (expressed is seconds) is the time needed to fill a 1 litre container placed under the cone were the grout was introduced. The environment conditions of the laboratory were characterized by 65% of RH and temperature of 21±1 ºC. The test for time of efflux was made immediately after grout preparation (Fig. 7.3).

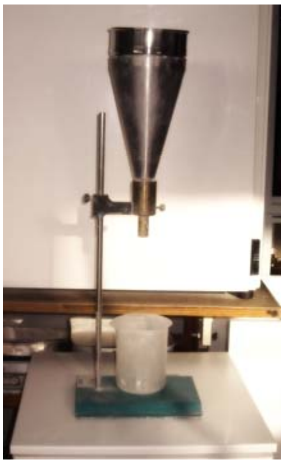

Figure 7.3: Cone test device according to EN445.

7.4.4 Rheological measurements

The rheological properties of the different grouts were studied using a Bohlin Gemini HR^{nano} rotational rheometer, in a plate/plate geometry (ϕ = 40mm), with a gap of 2mm. The samples were subjected to a pre-shearing stage during 30s at $\dot{\gamma} = 10s^{-1}$, 8 minutes after binder was brought in contact with water. After 60s at rest, a step up test of shear rate was applied including a linear 10-min upwards ramp from 0 to 300s^{-1}, 60 s at maximum shear rate and an analogous step-down from 300 s^{-1} to rest. The time step adopted was necessary to obtain the steady state behaviour for each shear rate applied.

Since a linear relationship was detected between shear stress and shear rate, after a yield stress value, a Bingham behaviour was assumed for each grout tested. In this way, two important values were taken for each grout sample: yield stress and plastic viscosity.

7.4.5 Injection tests

In order to study the grout injection capacity some tests were made. The aim of the laboratory research on grouts for injection is to define a right composition for grouts to be used for the strengthening of walls and to improve the knowledge about the physical mechanisms that take place during injection. Even if injectability tests are carried out on site, the characteristics of the grouts should be fine tuned in laboratory.

It is difficult to use samples of real masonries in lab conditions, not only on account of reproducibility issues, but also because it is impossible to visualize what is happening inside the porous media being injected by grout. Therefore the use of transparent Plexiglas cylinders was considered. These cylinders, with 144 mm of diameter and 300 mm height, were filled with a fraction of finely crushed limestone and injected unidirectionally from bottom to top (simulating masonry samples) (Fig. 7.4). This test was based on previous works of Toumbakari, Van Rickstal and Binda (Toumbakari, 2002), (Van Rickstal, 2000), (Binda *et al.*, 2003).

The cylinders were filled with either “2-4”mm or “4-10”mm aggregate and injected with the fresh natural hydraulic lime grouts immediately after grout preparation. The main goals were the following:

1) Check the minimum w/b ratio for the grout to be injectable;
2) Understand the grout flow inside porous medium for different w/b adopted;
3) Observe the relationship between injection time and flow speed for different w/b ratio used;
4) Analyse the previous results for different injection pressures (1.0bar, 1.5bar and 2.0bar).

A comparison of injection performance in the two types of porous media (“2-4”mm and “4-10”mm) was made for a constant pressure of 1.0bar.

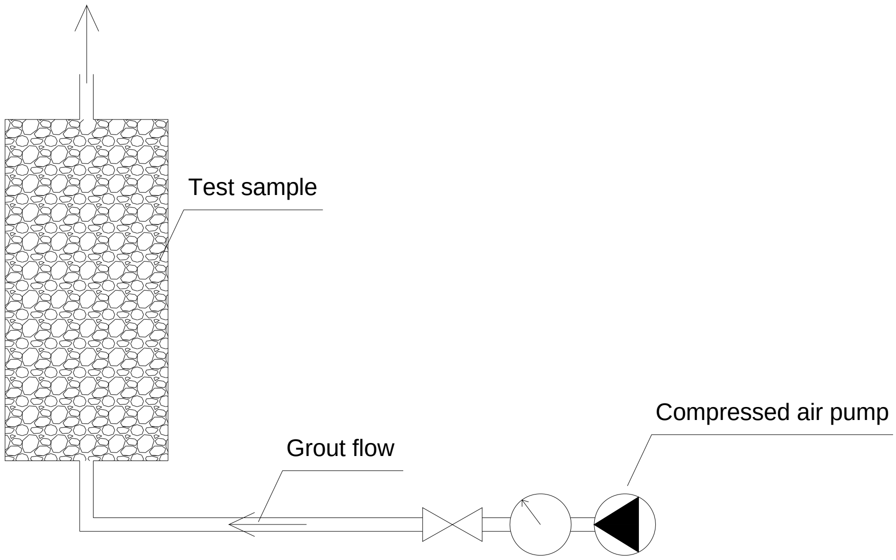

Figure 7.4: Setup for injection tests used in lab.

7.4.6 Injection tests for injection curves determination

Several cylinders were injected with water, NHL5 based grout (w/b=0.70) with or without 15% of fly ash (the best grout composition according rheological and strength results in Chapter 5).

An attempt to get an injectability curve for each grout tested as a function of the percentage of total mass that passes through the fine grained soil was also made. The goal was to get the grout injection capacity curve of different grouts in a given porous media. Five different sands were placed inside the cylinders: “0-4”mm; “0-2”mm; “0-10”mm; “2-4”mm; “4-10”mm (see Fig. 7.2). Each sand is characterized by a different percentage that passes through nº 40 sieve (0.425mm). So, different grout injection times will be obtained for coarse or fine grained porous media and different curves will be obtained for different grouts. For each porous media inside cylinders porosity was also measured.

There is no quantitative definition for injectability (I). However, it is known that it is higher for a small injection time (t) and that the injection efficacy is higher if the total height (h_{inj}) to be injected is fully filled by grout. Thus, the following expression was proposed:

$$I = \frac{1}{t} \times \frac{h_{inj}}{0.30} \tag{7.1}$$

where,

I = grout injectability (s^{-1})

t =grout injection time (s);

h_{inj} = injection height (m);

0.30= cylinder height (m)

For each type of grout an injectability curve can be obtained and a decision about the best grout to adopt can be made. If it possible to get the porous media porosity from the interior of masonry to be injected, than an estimative of the grout injection capacity could me made and then choose the most suitable grout composition.

7.4.7 Splitting tests

The optimal grout composition from an injectability point of view (according to the results of the previous points 7.4.1 to 7.4.5) was tested in its hardened state using the splitting test. For that purpose, the grouts were injected in porous media “2-4”mm type. Only one sample was tested for each different situation.

After 45 and 120 days, the injected cylinders were subjected to the splitting test in order to define the tensile strength of the “repaired” material. The indirect tensile strength test was carried out according to the (ASTM C 496/C 496M-04). This test method consists of applying a diametral compressive force along the length of a cylindrical concrete specimen at a rate that is within a prescribed range until failure occurs (Fig. 7.5). This loading induces tensile stresses on the plane containing the applied load and relatively high compressive stresses in the area immediately around the applied load. Tensile failure occurs rather than compressive failure because the areas of load application are in a state of triaxial compression, thereby allowing them to withstand much higher compressive stresses than would be indicated by a uniaxial compressive strength test result. The tensile strength value is calculated in the theoretical failure section where the tensile stress has the maximum value:

$$T = \frac{2P}{\pi \times l \times d} \qquad (7.2)$$

where:

T = splitting tensile strength (MPa)

P = maximum applied load indicated by the testing machine (N)

l = length (mm)

d = diameter (mm)

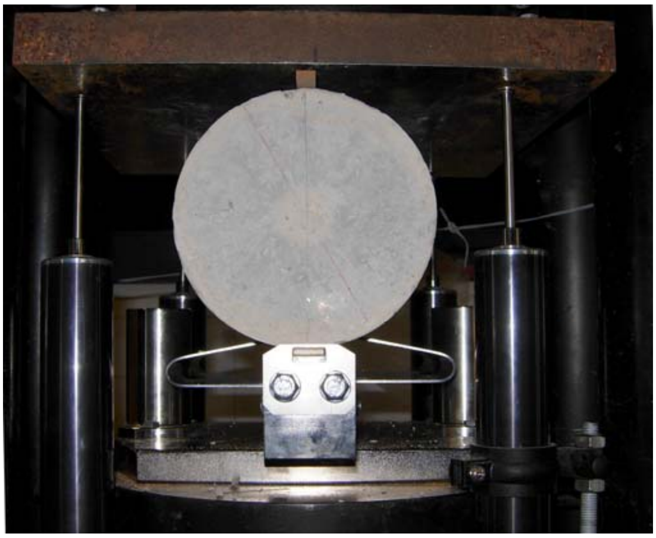

Figure 7.5: Splitting test device adopted.

7.5 Results and discussion

7.5.1 Bleeding test

The results obtained are presented in Fig.7.6.

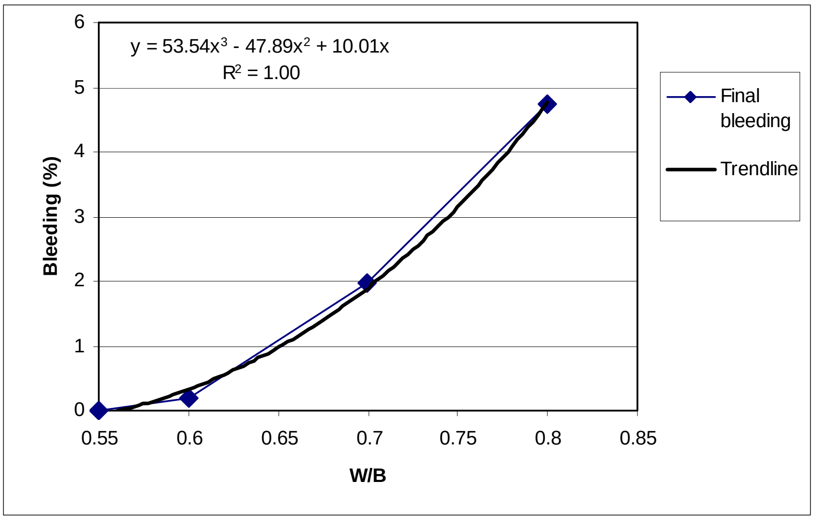

Figure 7.6: Bleeding for different w/b ratios tested for a natural hydraulic lime (NHL5) based grout.

According to Toumbakari and Mirza *et al.* (Toumbakari, 2002), (Mirza *et al.*, 2002) a grout has a good behaviour if the bleeding is less than 5%. Thus, all grouts are stable if $w/b \leq 0.80$.

This test was adopted to define the appropriated w/b range for the following tests ($0.55 \leq w/b \leq 0.80$).

7.5.2 Marsh cone

Flow times for the natural hydraulic lime grouts with different w/b ratio were determined and the results are presented in Fig. 7.7.

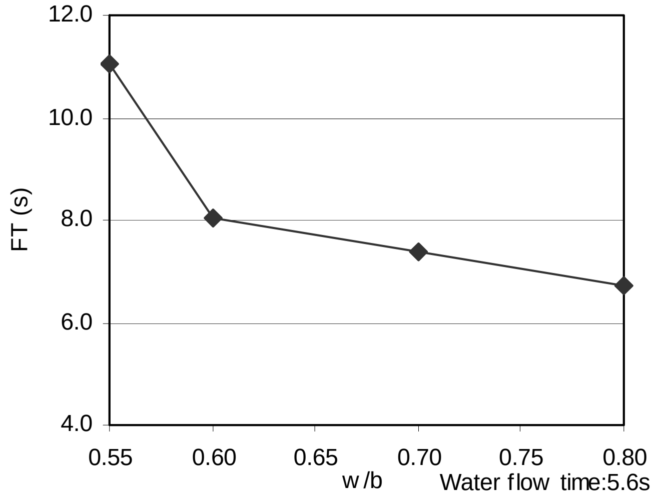

Figure 7.7: Flow time for 0 minutes after grout preparation for different w/b tested.

The analysis of the results shows that for $w/b \geq 0.60$ the flow time value is almost constant. It means that, from this point of view, there are no benefits if w/b is increased beyond 0.60.

7.5.3 Rheological measurements

Since Bingham behaviour was adopted, two parameters for rheological properties may be obtained: plastic viscosity and yield stress. Yield stress and plastic viscosity values for natural hydraulic lime (NHL5) grout *versus* different w/b tested are presented in Fig. 7.8. The rheological parameters were calculated from the downward curve (going from a higher degree of dispersion to lower one) since this is what happens when grout is injected inside masonry - at the end it stops to flow.

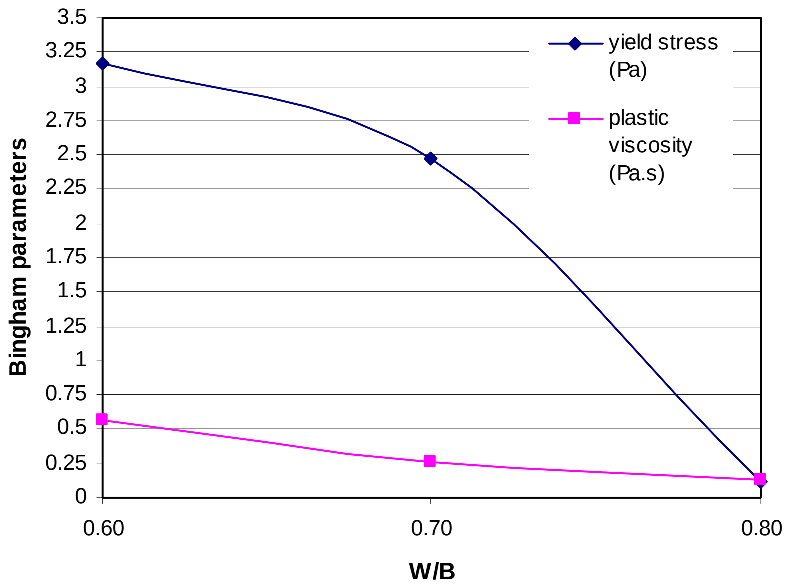

Figure 7.8: Plastic viscosity and Yield stress values for NHL5 grout versus different w/b (obtained from the downward curve).

According to Fig. 7.8, there is a considerable decrease of plastic viscosity in the range $0.60 \le w/b \le 0.70$ which is a good result since plastic viscosity is related with better grout injection capacity. A considerable decrease of yield stress was detected for $w/b \ge 0.70$. An increase of the w/b ratio decreases this parameter, which means that injection and penetration capacity will be better for $w/b \ge 0.70$.

Based on Figs. 7.7 and 7.8 it can be observed that flow time (FT) does not decrease too much for w/b>0.60. However, plastic viscosity and yield stress are still decreasing considerably until w/b=0.80. Thus, the best water/binder ratio for an optimal injection should be the one that simultaneously meets the following conditions: less bleeding (w/b must be less than 0.80), less plastic viscosity ($w/b \ge 0.70$) and low yield stress value ($w/b \ge 0.70$).

According to the previous results of bleeding, yield stress, plastic viscosity and flow time, the parameters which seem to be more conclusive in the choice of the best w/b ratio are the rheological ones. There are considerable changes in the rheological behaviour for $w/b \ge 0.70$.

However, flow time (a parameter generally used *in situ*) does not change much between $0.60 \le w/b \le 0.80$. This situation leads to an important doubt: is the flowability standard appropriated to grout design for masonry injection purpose?

7.5.4 Injection tests

For the cylinders filled by the fraction of crushed limestone type “2-4”mm (see grain size distribution in Fig. 7.1) the minimum w/b ratio allowing injectability was determined. It was found that for a $w/b \leq 0.55$ the grout was not injectable in the porous medium, so only $w/b \geq 0.60$ were considered.

The relation between the w/b ratios and injection time of the cylinders is plotted in Figure 7.9 for different injection pressures. The maximum water content was only 0.80 since grout bleeding should be limited (see 7.5.1 Bleeding test).

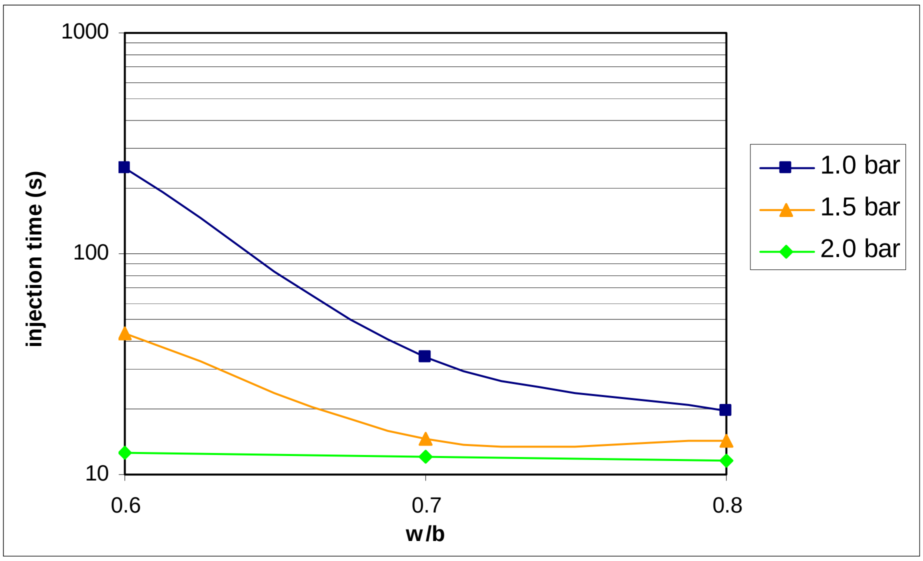

Figure 7.9: Average of injection times in “2-4”mm media of grouts with different water content and injection pressures.

The results obtained show differences when the water content and adopted pressures were altered. As it may be seen, there is an impressive decrease of injection time for $w/b \geq 0.70$, mainly for 1.0 bar and 1.5 bar. This means that the grout penetration capacity is optimized for that water content.

According to Bras and Henriques (Bras *et al.,* 2008) the analysis of flow time results shows that for $w/b \geq 0.60$ that value is almost constant. However, the analyses of injection time show different conclusions. There is indeed some difficulty in the injection of a grout with w/b=0.60 using a pressure of 1.0 or 1.5 bar and the flow inside the porous media is heterogeneous, which leads to an incomplete filling of the samples (Fig. 7.10 and 7.11).

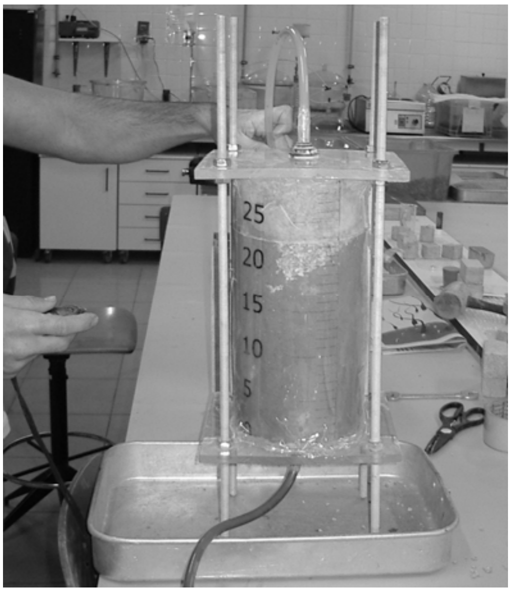

Figure 7.10: Cylinder filled by sand type “2-4”mm and injected with a pressure equal to 1.0 bar by NHL5 based grout with w/b=0.60. Injection being processed- non homogeneous filling.

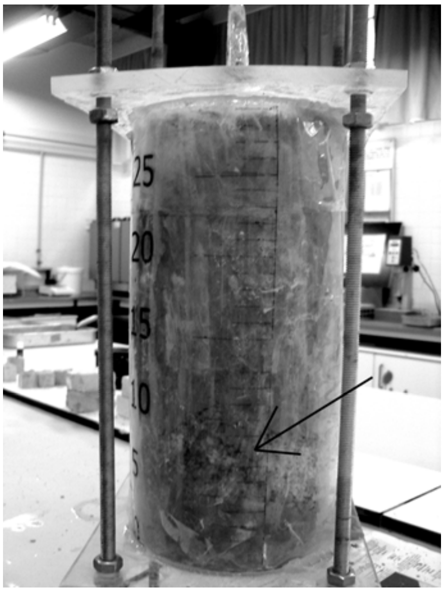

Figure 7.11: Cylinder filled by sand type “2-4”mm and injected with a pressure equal to 1.0 bar by NHL5 based grout with w/b=0.60. Porous media not injected was detected inside cylinder.

Nevertheless, for a w/b=0.70 the filling is almost homogeneous and the flow is uniform, as it can be observed in Fig. 7.12 and 7.13. The same results were detected for w/b=0.80.

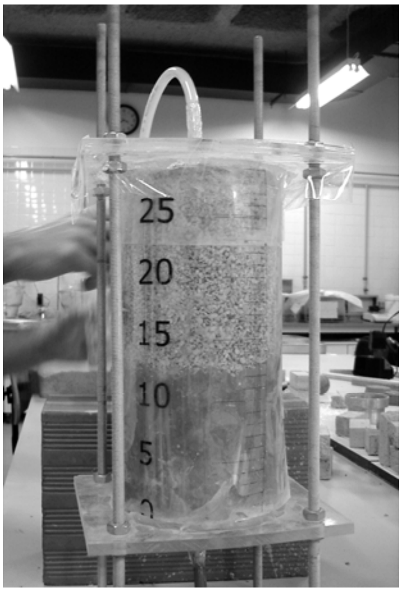

Figure 7.12: Cylinder filled by sand type “2-4”mm being injected by grout with w/b=0.70. The flow is uniform.

Figure 7.13: Grout with w/b=0.70 being injected in a cylinder filled by sand type “2-4”mm. The injection filling is almost complete.

As it can be observed in the previous figures, there is a considerable decrease of injection time for $w/b \geq 0.70$ which is not consistent with the flow time results. Only for larger water contents the injection times became almost constant (even for higher injection pressures). The analyses of the rheological behaviour of grouts lead to the same conclusion (see Fig. 7.8) - rheological grout parameters still decrease for $w/b \geq 0.70$ (and are not constant for $w/b \geq 0.60$ as it seems to occur with FT) - which proves the influence and importance of these properties in grout design and injectability, as confirmed by the injection tests.
The Marsh cone test seems to be inadequate for injection grout design, at least for this (or smaller) grain size distribution of the porous media.

In a general way, an increase of the injection pressure leads to the decrease of the time needed to fill all injected sample. However, there is an asymptotic value of time that was not changed even for greater injection pressures. It also seems that there is a threshold w/b ratio beyond which no benefits were detected in terms of injection efficacy. Fig. 7.14 presents the evolution of grout flow velocity inside the porous media for different w/b ratios and injection pressures.

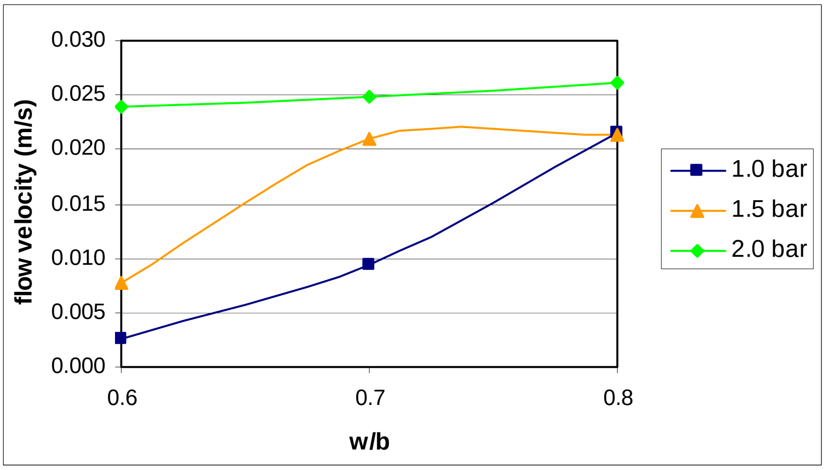

Figure 7.14: Average of flow velocity for NHL5 based grout with different water content, injected in the porous media "2-4"mm with different injection pressure.

An increase of the w/b ratio and injection pressure leads to an increase of the flow velocity. For 1.5 bar the velocity becomes almost constant if $w/b \geq 0.70$. For 2.0 bar, velocity does not change significantly. Velocity tends to an asymptotic value if water content is increased. The same happens if pressure is increased until 2.0 bar. This probably means that there is a threshold of injection pressure above which no benefits are obtained.

For each type of porous media the existence of an optimal injection pressure can be assumed. In this case, the pressure should not probably overpass 1.5 bar, also to insure masonry safety.

After the previous tests, the cylinders were filled with crushed limestone type "4-10"mm and re-tested. A comparison between the two types of porous media ("2-4"mm and "4-10"mm) was made for a constant pressure of 1.0 bar (the smallest adopted in this test). The main goal was to check the evolution of injection time and compare it with the flow time curve (Fig. 7.15).

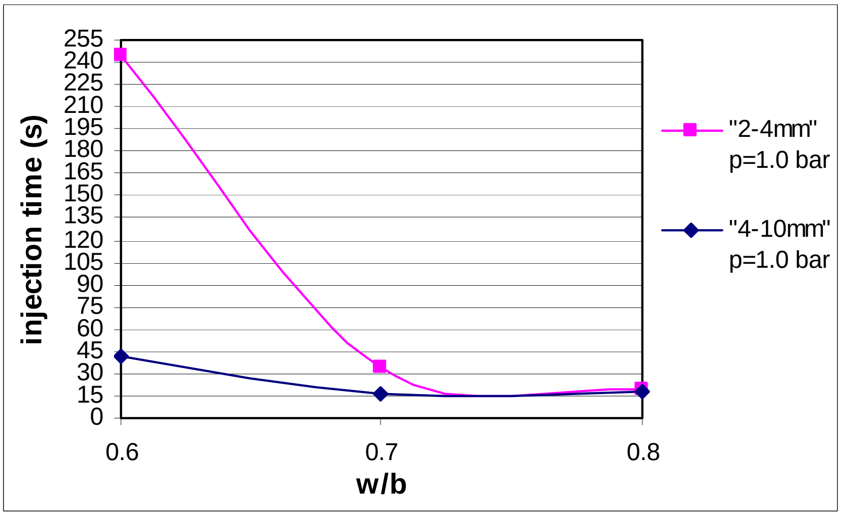

Figure 7.15: Comparison of injection time values for NHL5 based grout with different water content, injected in the porous media “2-4”mm and “4-10”mm with p= 1.0 bar.

Considerable differences of injection times were detected for w/b=0.60 and w/b=0.70 for the finer aggregate, which are almost negligible for the coarser media.

Comparing these results with those obtained for the flow times with the Marsh cone (Fig. 7.7) it appears that the Marsh cone test may be applicable for grouts to be used in masonries with larger voids (tentatively equal or greater than 4mm). This indicates that the grain size distribution of the porous media to be injected has an important influence on the type of analysis that should be performed. The Marsh test can only be adopted for a certain grain size distribution of masonries; in these situations it will become an effective and quick test to apply on field for grout design and for controlling the constant mixing performance.

According to the previous results, it seems that the optimum solution for a better injection capacity leads to a natural hydraulic lime based grout with $w/b \geq 0.70$. For w/b=0.70 the grout flow is uniform and homogeneous inside the porous media even for an injection pressure of 1.0 bar. An increase of the injection pressure enables injection time reduction; however it may also lead to masonry safety problems, namely from strength and durability points of view. Thus, the selection of the best pressure should always take into account this aspect, meaning that in the analysed range p=1.0 bar is the best option. Since water content higher than 70% leads to a less visible improvement of the injectability properties and reduces the grout strength, the w/b ratio should be the minimum possible to obtain an appropriate injectability.

Real masonry is heterogeneous and has always zones with very different porosity. Among that, the coarse or the fine zone is not always the most endanger one. Thus, the grout criteria selection should always take into account this important variable, knowing that there are always limitations in the methodology.

Since sometimes it is possible to obtain a grain size distribution of the interior of a masonry to be injected, then the injection capacity of a grout can be estimated and the most suitable grout composition chosen accordingly. For each type of grout an injectability curve can be obtained and a decision about the best grout to adopt can be made. Tests have been made by the authors for injectability curves determinations. Those studies are not shown in the present work in spite of its relevance for injectability purposes, although it will be considered in future developments.

Eklund and Stille (Eklund *et al.*, 2008) state that the ability of a grout to penetrate cavities, channels and porous material (injection capacity or penetrability) depends on two aspects: rheology and filtration tendency. Filtration tendency is the ability of a grout to prevent passing obstructions in the flow path without the binder grains clogging and preventing further penetration. This property of a grout is the property that defines its ability to form a plug in a crack or during entry into it and that behaviour is due to the fact that binder grains are in more or less constant contact with each another. For grouts with low w/b ratio the contact of grains is similar to the one of dry binder.

According to some experiments performed by these authors with aggregate and cement material, it seems to be advantageous for penetrability to have a grain-size distribution that does not contain too many fine or coarse grains. Too large maximum grain-size will obstruct the flow, preventing penetration of the grout. The maximum grain-size may be characterized for example in terms of d_{95} (the diameter through which 95% of the total binder mass is passing). According to the referred tests the value of d_{95} should be 4–10 times less than the aperture to be penetrated by the cement-based mixture.

The binder used for this work has a d_{95} less than 0.2mm (Chapter 6). Since the average dimension of voids inside the porous media is 2.7mm in “2-4”mm type and even greater in “4-10”mm type, it is reasonable to assume that the previous rule is verified, thus avoiding possible obstruction of the flow path.

However, as it can be seen in Fig. 7.10, in some sections the flow path had not the same evolution in terms of grout filling, probably due to voids dimension variation inside the porous media. This phenomenon is not exclusive for grouts with w/b=0.60, it could also happen for higher ratios.

Low w/b ratio grouts show that the most significant limitation to penetrability is the tendency of binder grains to agglomerate into an impermeable filter cake. A small amount of binder fines is also important in achieving low filtration tendency of the grout because of the increased tendency for small grains to flocculate into larger agglomerates, when compared to larger particles. The filtration experiments with cement based grouts show that parameters such as cement chemistry (hydration of cement grains) will strongly affect the filtration tendency of the mixture (Eklund *et al.*, 2008). This statement highlights the importance of the hardening time of grouts and its injection capacity.

According to some previous works regarding rheology of fresh grouts (Roussel, 2007), (Eriksson *et al*., 2000), from a practical point of view yield stress is probably associated to the ability of the grout in filling up the voids and if it is able to flow when a shear stress is applied. The knowledge of the yield stress enables the understanding if a fluid will flow or not, since it represents that threshold. This important property affects the flow behaviour and the capacity to flow inside a porous medium, controlling the maximum penetration length that grout can reach inside masonry. Thus, yield stress may be used as a control index for the application of injection grouts.

The comparison between yield stress (Fig. 7.8) and injection capacity of the tested grouts emphasize the influence of this rheological property in grout behaviour. A greater yield stress leads to a reduction of injection capacity, as it can be observed when a small water content is used in grout composition (w/b=0.60).

Supposing that grout flow occurs inside a void with the shape of a cylindrical tube and that the flow obeys to the following conditions:

a) the flow is laminar (which is true in our tests);
b) grout flow does not change in time;
c) no shear movements occur at the void wall;
d) shear rate in one point is only function of the shear in that point (Fig. 7.16).

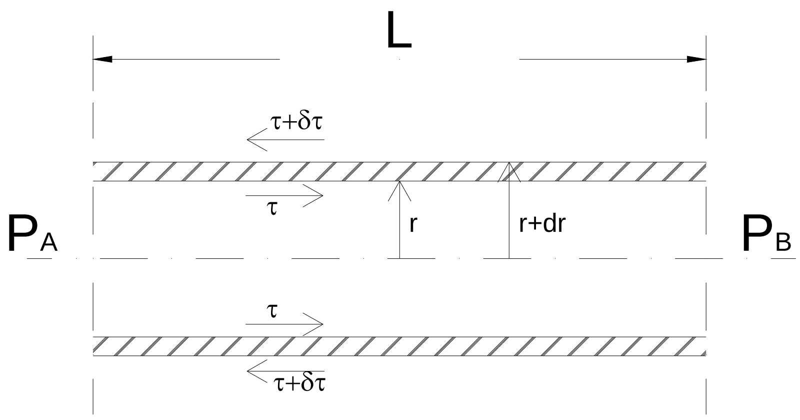

Figure 7.16: Void with the shape of a cylindrical tube were flow occurs from A to B.

where:

τ =shear stress at void wall

$\delta\tau$ =shear stress increase

r =void radius

δr =radius increase

P_A =injection pressure in point A (flow goes from A to B)

P_B =injection pressure in point B

According to Buckingham Reiner equation, the shear stress at the wall of the cylindrical channel will be:

$$\tau = \frac{P_{A-}P_B}{L} \times \frac{r}{2} \Leftrightarrow \tau = \frac{\Delta P}{L} \times \frac{D}{4} \qquad (7.3)$$

where D is the dimension of the void.

Since the injection pressure is constant, the shear stress at the wall will decrease when the grout penetrates the channel because L, the length filled by grout, is increasing. When shear stress at the wall is lower than the yield stress of grout the flow will stops. This is expressed by the following expression:

$$\frac{\Delta P}{L} \times \frac{D}{4} \leq \tau_0 \qquad (7.4)$$

Knowing that the maximum L of the injection tests is 0.30m and the yield stress for each grout, the minimum void diameter (D_{min}) of the porous media to be injected can be estimated according to Eqn. 7.4. The worst condition of flow occurs for the smallest pressure adopted ($\Delta P = 1bar$). Table 7.3 presents D_{min} for each of w/b ratios considered.

Table 7.3: D_{min} of the porous media that could be injected for each w/b ratio adopted in grout composition.

w/b	Yield stress (Pa)	D_{min} (mm)
0.60	3.17	0.038
0.70	2.47	0.030
0.80	0.12	0.001

Analysing Fig. 7.1, for the grain size distribution type “2-4”mm the diameter through which only 1% of the total sand mass is passing is higher than D_{min} for w/b=0.60. However, the variability of voids inside porous media could also be a possible explanation to the difficulty that occurs when a w/b=0.60 grout is injected in that porous media, since D_{min} is greater in this case due to a high yield stress. Another possible explanation for the non homogeneous filling observed for grouts with w/b=0.60 may be the high water absorption of porous media (Fig. 7.17). The loss of water that occurs in grout leads to an increase of yield stress which means that Eqn. 7.4 may be satisfied- flow will stop in some channels- meaning that grout will try to flow through other available channels, hence the non homogeneous filling.

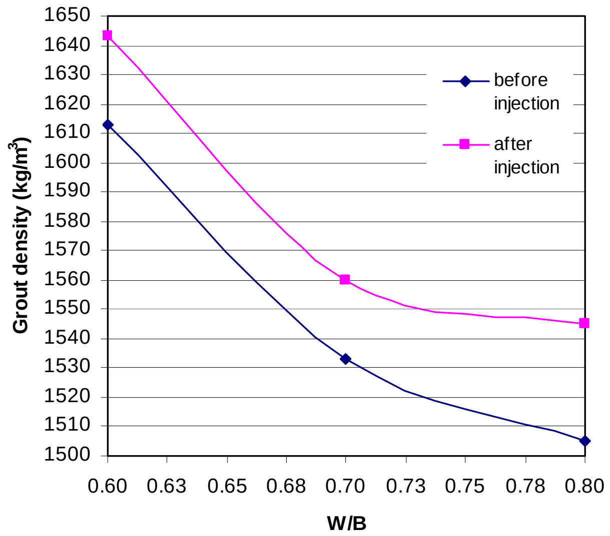

Figure 7.17: Grout density before and after injection in the porous media type “2-4”mm with p=1.0 bar.

The density increase is associated to porous media water absorption, meaning for instance that an initial w/b=0.70 will became a grout with w/b=0.66 at the end for injection (>yield stress).

Plastic viscosity is another rheological property that may be associated to the velocity at which a given grout will flow once flow begins. Like yield stress, plastic viscosity may also work as a prediction factor of whether a grout will be able to be pumped (Roussel, 2007). The comparison of viscosity behaviour (Fig. 7.8) and the flow velocity inside the cylinders (Fig. 7.14) indicates that for a grout with less viscosity its flow velocity is improved. However, this behaviour tends to disappear if the injection pressure is increased.

Above a given injection pressure, flow in the porous medium is fast enough for inertia effects to play a role and prevent the injection speed to increase further. Once again, this indicates that Marsh cone test accuracy is smaller for use it in grout mix design if higher injection pressures are adopted for masonry consolidation. The analyses of the rheological behaviour of grouts, especially plastic viscosities, lead to the same conclusion: it became constant only for w/b=0.70 (and not for $w/b \geq 0.60$) - which proves the influence and the importance of these properties in grout design and injectability, as confirmed by the injection tests.

Besides that, for low viscosity grouts such as those used, inertia effects start to play an important role and Marsh cone flow times are no longer proportional to viscosity. In these cases, the diameter of the cone nozzle should be decreased if a rheological correlation is expected. This is in agreement with the findings of Roussel and Le Roy (Le Roy *et al.*, 2004), (Roussel *et al.*, 2005).

7.5.5 Injection tests for injection curves determination

After the selection of the best injection pressure and w/b ratio for a NHL5 grout, the same procedure was adopted for a NHL5 + 15% fly ash based grout. An injectability curve was obtained for NHL5 grout and NHL5 +15% of fly ash based grout with w/b=0.70, using the procedure described in 7.4.6. The water content adopted was the optimal obtained according to the previous results of this chapter.

The five different porous media were injected using a pressure of 0.7 bar. This pressure was selected since the behaviour for 1.0bar was really good, meaning that it was still possible to reduce injection pressure (taking also into account Eqn. 7.4). Table 7.4 presents the % of the total mass that passes through nº 40 sieve for each porous media placed into cylinders, the voids size average injected, as well as its porosity. Fig. 7.18 and 7.19 present the injectability values together with the respective trend lines for water, NHL5 and NHL5+ 15% fly ash with w/b=0.70.

Table 7.4: Porous media characteristics.

Porous media type	"4-10"	"2-4"	"0-10"	"0-4"	"0-2"
% of the total mass that passes through nº 40 sieve	0.0%	7.4%	18.20%	21.3%	30.0%
Porosity (%)	45	40	28	23	13.5
Voids size average (mm)	4.16	2.87	1.54	1.2	0.65

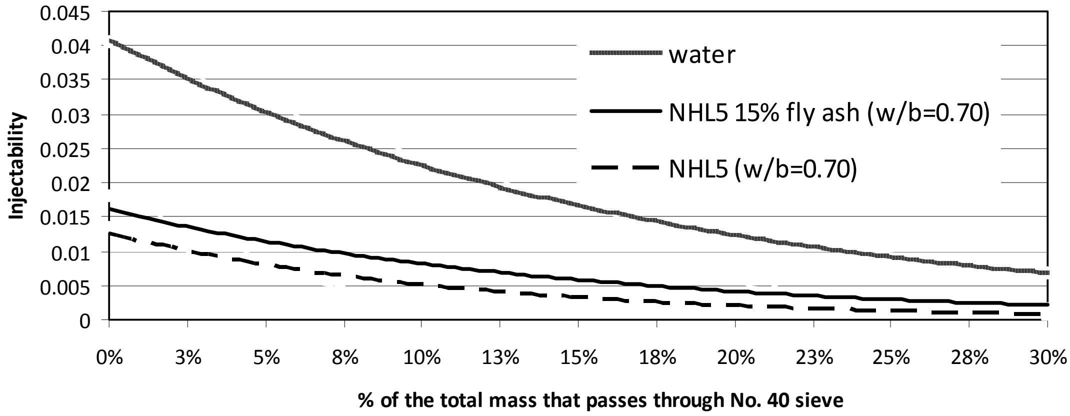

Figure 7.18: Injectability curves for water, NHL5 and NHL5+15% fly ash for the different porous media tested, taking into account the % of the total mass that passes through nº40 sieve (0.425mm).

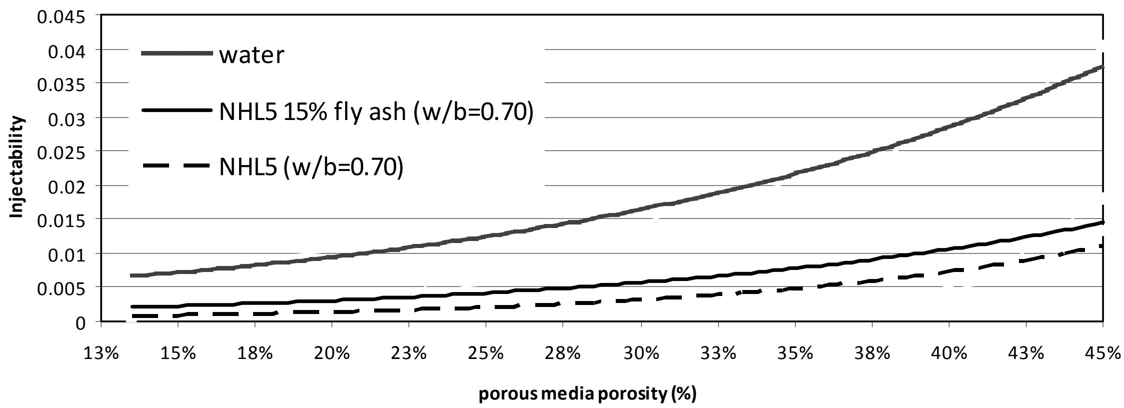

Figure 7.19: Injectability curves for water, NHL5 and NHL5+15% fly ash for the different porous media tested, taking into account the porous media porosity injected.

Water curve correspond to the best behaviour in terms of injection capacity and the addition of fly ash improve that capacity. Injectability becomes dimensionless if I is divided by I_{water} (water injectability). Fig. 7.20 and 7.21 presents grout injectability in terms of % of I_{water}.

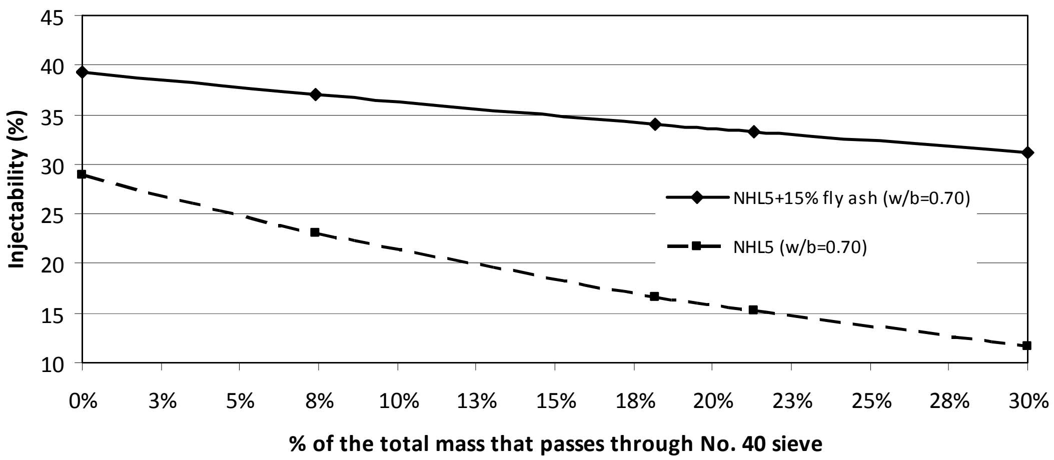

Figure 7.20: Dimensionless injectability curves for NHL5 and NHL5+15% fly ash based grouts.

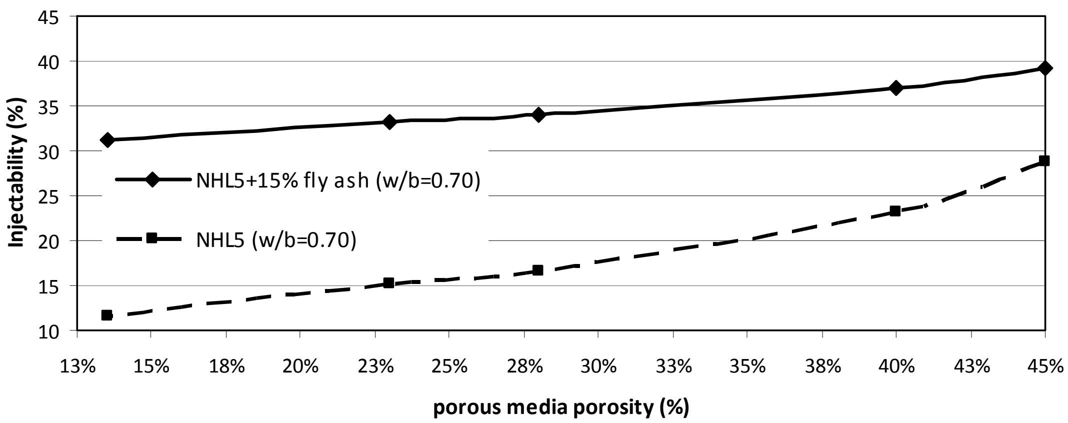

Figure 7.21: Dimensionless injectability curves for NHL5 and NHL5+15% fly ash based grouts for different media porosity.

From the previous figures it can observed that injectability is quite different for the two tested grouts. However, when comparing it with water results (Fig. 7.21) it seems that grout injection capacity is to below at least for the low porous media.

According to (Laefer *et al.*, 1996) multiple leaf walls can be made with very poor mortars and stones. If they have very low percentage of voids (Fig. 7.22) (less than 4% of voids) they will not be injectable.

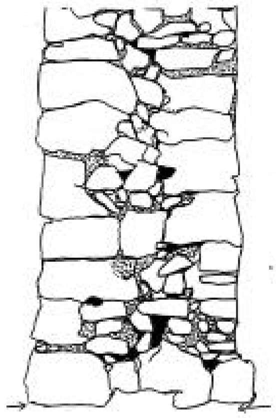

Figure 7.22: Poor section with very low content of voids (black filled) (Laefer *et al.*, 1996).

Other researchers, such as Binda, have studied masonry walls of buildings and tried to calculate its voids (Binda *et al.*, 1993), (Binda *et al.,* 1997), (Binda *et al.*, 2001), (Penazzi *et al.*, 2001). The studies were carried out in different Italian regions. The survey consisted of a graphic and photographic procedure which included taking a photograph with a camera having the lens of 50 mm using a tripod which ensures the parallelism between the plane of the photograph and that of the wall; working out the metric survey of the walls first manually and then on a PC; creating a rich data-base organized in tables. The survey of the internal sections allowed to define some important parameters like: the percentage distribution of stones, mortar, voids which allows to make comparisons between the different regions (Fig. 7.23 and 7.24).

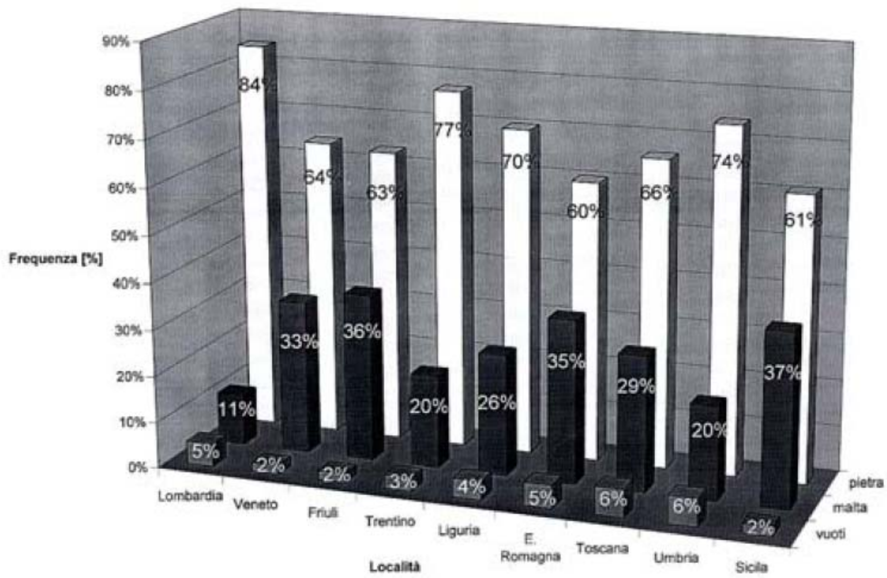

Figure 7.23: Percentage of mortar versus percentage of stones referred to the area of the cross section of stonework walls in various Italian regions (Binda *et al*., 2001).

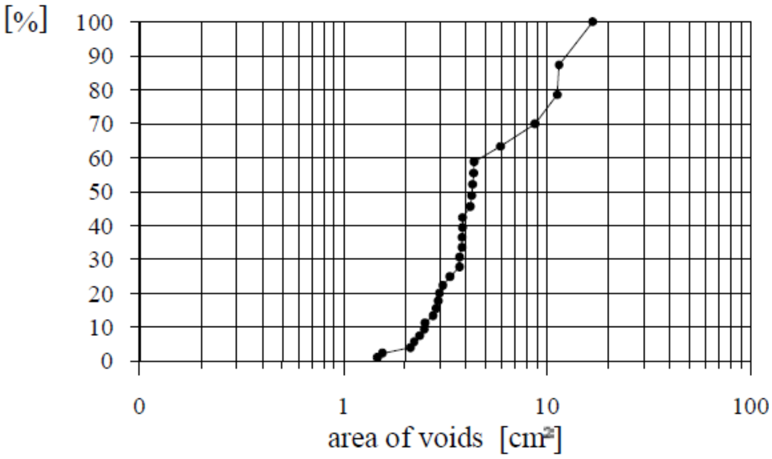

Figure 7.24: Size and distribution of the voids within the section of the wall (Binda *et al.*, 1997).

The void diameter average found as example (Fig. 7.24) is 2.3cm and 90% of it is higher than 1.7cm, meaning that the methodology proposed in this Chapter 7 for testing grout injectability in a porous media like masonry is a logical tool to use in grout optimization (see Table 7.4).

Some similar testes have been made in Portugal, trying to characterize masonries sections, namely by Department of Civil Engineering of UNL. However, that survey is still beginning and no results are presented here.

7.5.6 Splitting tests

Indirect tensile strength of the injected cylinders with the optimal grout composition is presented in Table 7.5.

Table 7.5: Indirect tensile test on injected cylinders at age 45 and 120 days for NHL5 and NHL5+15% fly ash (w/b=0.70).

Specimen	Diameter(mm)	Length (mm)	T (MPa)
$NHL5_{45d}$	143	297	0.45
(NHL5+ 15% fly ash)$_{45d}$	144	300	0.50
$NHL5_{120d}$	142	290	0.76
(NHL5+ 15% fly ash)$_{120d}$	142	295	0.73

Splitting test enables the determination of grout bond capacity to the porous media injected. Only a grout with good behaviour will improve the load bearing capacity of masonry.

As it can be observed, at 45 days of age there are no main differences between the grout with or without fly ash, so it is difficult to get a safe conclusion. However, the small strength increase detected for NHL5+ 15% fly ash comparatively to NHL5 based grout probably means that this grout seems to behave better, which is also in agreement with the results obtained in Chapter 5 for grouts individual samples. Thus, the addition of fly ash tended to increase strength, which is in agreement with Toumbakari (Toumbakari, 2002). Probably fly ash reduce porosity and increase densification of interfacial bond between aggregate and grout, explaining the previous results (Chapter 5). SEM results of the samples would help in these confirmations.

Despite the differences in the porous media tested, those tensile values (0.45MPa and 0.50MPa) are in the same magnitude has in (Binda *et al.*, 2003) meaning the reproducibility of this test.

120 days after grout injection it can be detected an improvement of grout bond capacity, probably mainly due to continuous NHL5 hydration and/or calcium hydroxide consumption by fly ash to form silicates and aluminates. Tensile strength increased almost 1.7 times. However, no significant difference was observed between the two types of grout tested.

7.6 Main conclusions

Analyses of some relevant grout properties such as yield stress, viscosity, bleeding, flowability and injectability capacity for natural hydraulic lime based grouts (EN459-1 NHL5) was made. It shows that there are substantial variations on grout properties as a consequence of different water/binder ratios (w/b) and try to increase the understanding of the influence of the mixing design in grouts behaviour and how it may improve some essential injection characteristics. For that, injection tests in cylindrical samples trying to reproduce masonry materials were made. An attempt was also made to achieve an optimal solution for grout design. The study has envisaged to increase the knowledge about the physical mechanisms that take place during injection and to define a right composition for grouts to be used for the strengthening of walls. The main conclusions are the following:

- According to the previous results of: bleeding, yield stress, plastic viscosity and flow time, the parameters which seems to be more conclusive in the choice of the best w/b ratio are the rheological ones. Flow time (a parameter generally used *in situ*) does not change much between $0.60 \leq w/b \leq 0.80$. It means that, from this point of view, there are no benefits if w/b is increased beyond 0.60. However, the analysis of rheological parameters shows that there is a considerable decrease of plastic viscosity until w/b reaches 70%, after which that parameter does not change that much and yield stress value decreases almost 1.5 times if the w/b ratio goes from 60% to 70% and 20 times when w/b ratio goes from 70% to 80%.

- During cylinders injection only grouts with $w/b \geq 0.60$ may be injected in a porous media type "2-4"mm.
- The results obtained in that porous media show differences for different water content and pressure adopted during grout injection. As it may be observed, there is an expressive decrease of injection time for $w/b \geq 0.70$, mainly for 1.0 bar and 1.5 bar. This means that the grout penetration capacity is optimized for that water content.
- There is indeed some difficulty in the injection of a grout with w/b=0.60, using a pressure of 1.0 or 1.5 bar, and the flow inside the porous media is heterogeneous, which leads to an incomplete filling of the injected samples with grout. Only for $w/b \geq 0.70$ the grout filling becomes homogeneous and uniform.
- The injection time value became almost constant for $w/b \geq 0.70$, even for an increasing injection pressure. The analyses of the rheological behaviour, especially plastic viscosity values (which are related with flow velocity), of grouts lead to the same conclusion: it became constant only for that w/b range (and not for $w/b \geq 0.60$) - which proves the influence and the importance of these properties in grout design and injectability, as confirmed by the injection tests.
- The Marsh cone test seems to be inadequate for grout design for injection purpose at least for this grain size distribution (type "2-4"mm) of the porous media, or even smaller- as it was demonstrated in this paper- when FT was compared to grout rheological parameters and to injection values. Besides that, for low viscosity grouts such as those used, inertia effects start to play an important role and Marsh cone flow times are no longer proportional to viscosity. In these cases, the diameter of the cone nozzle should be decreased if a rheological correlation is expected.
- An analysis regarding an higher grain size distribution of the porous media (type "4-10"mm) indicate that the grain size distribution of the porous media to be injected has an important influence on the type of analysis that should be made in grout design for consolidation purpose: Marsh cone device should be modified and adopted for a certain grain size range of masonry in order to become an effective and quick test to apply on field for grout design.

- Probably, if there is a correlation between experimental results of rheological parameters and flow time (which is not true in a porous media type “2-4”mm), then a mathematical expression for flow time, yield stress and plastic viscosity will be possible and will be very useful for grout design for injection purpose.
- According to the previous results (7.5.1 to 7.5.4), it seems that the optimum solution for a better injection capacity leads to a natural hydraulic lime based grout with w/b=0.70. In this situation the grout flow is uniform and homogeneous inside the porous media even for an injection pressure of 1.0 bar. An increase of the injection pressure enables injection time reduction; however it may also lead to masonry safety problems, namely in a strength and durability point of view. The selection of the best pressure should always regard the previous aspect and it means that, in the analysed range, p=1.0 bar is the best option.
- In order to avoid possible obstruction of the flow path inside masonry, the maximum and minimum binder grain size distribution should be controlled. The small amount of small grain size of a binder is also important in achieving low filtration tendency of the grout.
- The minimum void diameter (D_{min}) of masonry that could be injected by grout is significantly dependent on w/b ratio used in grout composition and on porous media water absorption capacity since it may increase grout yield stress.
- An attempt to get injectability curves of two different grouts was made (7.5.5). If it possible to get a grain size distribution from the interior of a masonry to be injected and put similar porous media in a device like 7.4.5, it in possible to estimate what is the injection capacity of a grout and choose the most suitable grout composition.
- Probably, fly ash reduce porosity and increase densification of interfacial bond between aggregate and grout, explaining the previous results of splitting test (7.5.6). However, more splitting test should be made. SEM results of the tested samples would help in these confirmations and more samples for splitting test should also be made.
- It is possible to highlight the importance of an optimal grout composition from a fresh and an hardened state.
- Real masonry is heterogeneous and has always zones with very different porosity. Among that, the coarse or the fine zone is not always the most endanger one. Thus, the grout criteria selection should always take into account this important variable, knowing that there are always limitations in the methodology.
- The survey of masonry internal sections allowing the definitions of some important parameters like: the percentage distribution of stones, mortar, voids is essential for

masonry characterization. Similar tests made in Italy should be adopted in Portugal trying to characterize masonries sections in all country.

- The methodology proposed in this Chapter 7 for testing grout injectability in a porous media like masonry is a logical tool to use in grout optimization.

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Chapter 8. Computational modeling for grout behaviour prediction

8.1 Purpose

According to the results presented in Chapter 7 for bleeding, yield stress, plastic viscosity and flow time, the parameters which seem to be more conclusive in the choice of the best w/b ratio for grout composition are the rheological ones. Flow time (a parameter generally used *in situ*) does not change much between $0.60 \leq w/b \leq 0.80$. It means that, from this point of view, there are no benefits if w/b is increased beyond 0.60. However, the analysis of rheological parameters shows that there is a considerable decrease of plastic viscosity until w/b reaches 70%, after which that parameter does not change that much and yield stress value decreases almost 1.5 times if the w/b ratio goes from 60% to 70% and 20 times when w/b ratio goes from 70% to 80%.

Thus, the device used for the Marsh cone test should be modified and adopted for a certain grain size range of masonry in order to become an effective and quick test to apply on field for grout design. A comparison between the numerical simulation and experimental results of Marsh cone was made. A simple approximation using Bingham behaviour was adopted. The goal was to get a good agreement between the numerical results and experimental values, in order to confirm the validity of the numerical approach and allow using those numerical results obtained for Marsh cone to predict grout flow time. An attempt to relate the rheological parameters of the tested grouts to their flow time through the cone was made.

8.2 Introduction

In terms of the physical phenomena to be modelled, the problem to be dealt with is not trivial. Grout testing of flow time in a Marsh cone can be described as a free surface flow of a non-Newtonian fluid. The flow is by nature transient. Among that, grout is constituted of particles and its behaviour is developing either reversibly (thixotropy) or irreversibly (hydration process). It is assumed that fresh grout behaves as a yield stress fluid and that the Bingham or Herschel Bulkley models are the most common models, but in order to choose this type of modeling, it is necessary to assume that grout can be considered a homogeneous single fluid

(Coussot et al., 1999), (Roussel, 2006), (Roussel *et al.*, 2007), (Martys, 2005), (Wallevik J., 2003).

In this chapter it will be presented the numerical simulation results of the flow in a Marsh cone test, obtained using the Computational Fluid Dynamic (CFD) solving algorithm FLUENT ©, to characterize the fluidity of hydraulic lime grouts. A comparison between the numerical simulation results of Marsh cone was made with the experimental ones. A simple approximation using Bingham behaviour was adopted. The goal was to get a good agreement between the numerical results and experimental values, in order to confirm the validity of the numerical approach and allow us to use those numerical results obtained for Marsh cone to predict grout flow time.

It will be shown that the flow time is directly linked to the grout rheological behaviour, namely yield stress and plastic viscosity parameters. Those results are in agreement with (Roussel *et al.*, 2005), (Nguyen *et al.*, 2006).

8.3 Gambit

GAMBIT is a software package designed to build and mesh models for CFD and other scientific applications like FLUENT. GAMBIT receives user input by means of its graphical user interface (GUI). The GAMBIT GUI makes the basic steps of geometry creation, meshing generation and assigning zone types to a model simple and intuitive (GAMBIT, 2004). GAMBIT mesh could then be exported to FLUENT 6 format where CFD is applied.

8.4 Fluent code

FLUENT-ANSYS version 6.3.26 applies the Finite Volume Method to solve continuity equation and Navier-Stokes equations. These Navier-Stokes equations describe how the velocity, pressure, temperature and density of a moving fluid are related. The equations were derived independently by G.G. Stokes, in England, and M. Navier, in France, in the early 1800's. The equations are extensions of the Euler equations and include the effects of viscosity on the flow. They are a set of coupled differential equations and could, in theory, be solved for a given flow problem by using methods from calculus.

The derivation of the Navier–Stokes equations (Eqn 8.1) begins with an application of Newton's second law: conservation of momentum (often alongside mass and energy conservation) being written for an arbitrary portion of the fluid. In an inertial frame of reference, the general form of the equations of fluid motion is:

$$\rho(\frac{\partial v}{\partial t} + v.\nabla v) = -\nabla p + \nabla\Gamma + f \tag{8.1}$$

Where v is the flow velocity, ρ is the fluid density, p is the pressure, Γ is the deviatoric stress tensor, f is the body force (per unit volume) acting on the fluid and ∇ is the del operator. This is a statement of the conservation of momentum in a fluid and it is an application of Newton's second law to a Continuum; in fact this equation is applicable to any non-relativistic continuum and is known as the Cauchy momentum equation (Batchelor, 1967).

In practice, these equations are too difficult to solve analytically so, approximations to those equations are necessary. Techniques like finite difference, finite volume, finite element and spectral methods are some options. CFD software (like FLUENT) deals with that.

In FLUENT code, the variables are defined in the center of each element. The diffusion terms are discretized using a second order differencing scheme and several resolution algorithms are available.

The VOF model can model two or more immiscible fluids by solving a single set of momentum equations and tracking the volume fraction of each of the fluids throughout the domain. This formulation relies on the fact that two or more fluids (or phases) are not interpenetrating. Thus, a variable is established if a new phase is introduced: the volume fraction of the phase in the computational cell. In each control volume, the volume fractions of all phases sum to unity. Detailed information could be found in (FLUENT, 2006).

In the study developed, the free surface modeling use VOF method, which identifies the free surface position using volume fraction index that could takes values from 0 (when the phase is air) to 1 (phase is the fluid-grout). Free surface position is arbitrary defined taking the value 0.5.

2D simulation was adopted in the model presented in this chapter. First order implicit was selected for the unsteady formulation and laminar flow used, since its precision was not so different from the second order formulation. The SIMPLEC algorithm, that allows coupling velocity and pressure, is used without sub-relaxation factors. The convective term at the face of control volume are calculated with the Second Order Upwind scheme, for the components of Navier-Stokes equations. In this approach, higher-order accuracy is achieved at cell faces through a Taylor series expansion of the cell-centered solution about the cell centroid. For the convection term of the volume fraction index, the scheme Modified HRIC (High Resolution Interface Capturing), specially developed for modeling free surface flow, is used. Finally the pressure is defined using the PRESTO! Scheme (PREssure STaggering Option) (FLUENT, 2006), (Paixão *et al.*, 2009).

8.5 Implementation of grout flow simulation in Marsh cone test

The flow of a Bingham grout was simulated in a Marsh cone according to EN445 (Fig. 7.3) using the commercial code FLUENT 6.3.26. The fluids presented various rheological characteristics: the yield stress varies from 0.34 to 2.13 Pa and plastic viscosity from 0.09 to 0.23 Pa.s. For all the studied grouts, the density of the fluid was measured. Those values are presented in Table 8.1.

Since a homogeneous approach was adopted, it means not only that the grout must be homogeneous but also that it stays homogeneous during flow. This was taking into account not choosing grout with bleeding effect ($w/b \leq 0.80$) (see Chapter 7). Thus, it was not necessary to introduce particles in the modeling (Coussot, 2005).

The experimental flow time is measured for a certain amount of the tested material. It is the time to flow and fill the vessel with 1liter.

Table 8.1: NHL5 based grout properties used in computational simulations.

w/b	0.6	0.7	0.8
yield stress (Pa)	2.13	0.91	0.34
plastic viscosity (Pa.s)	0.23	0.13	0.09
grout density before injection (kg/m^3)	1613	1533	1505

In order to reduce the numerical computational time only half cone was modelled, using a symmetry plan. As the Marsh cone is axisymmetric, it is enough to model the flow in a radial plane (2D).

In this radial plane, the fluid flows only under the effect of gravity and the free surface goes down during the flow (it was adopted the implicit body force). The problem can be calculated by using the VOF. This model is applied to the fixed mesh for two immiscible phases: air and a Bingham fluid. For each phase, the volume fraction (ratio between the volume occupied by the phase and the control volume) is introduced as a local variable and is calculated for each cell and at each moment based on the continuity equation and the momentum equation by taking into account the initial and boundary conditions of the flow. As the volume fractions of the phases vary as a function of the position and time, the free surface corresponding to the iso-surface of fluid (or air) volume fraction equal to 0.5 is moved.

Fig. 8.1 presents part of the Marsh cone mesh, generated with the software GAMBIT, adopted in the numerical simulations. The initials and boundary conditions of the flow are presented in Fig. 8.2.

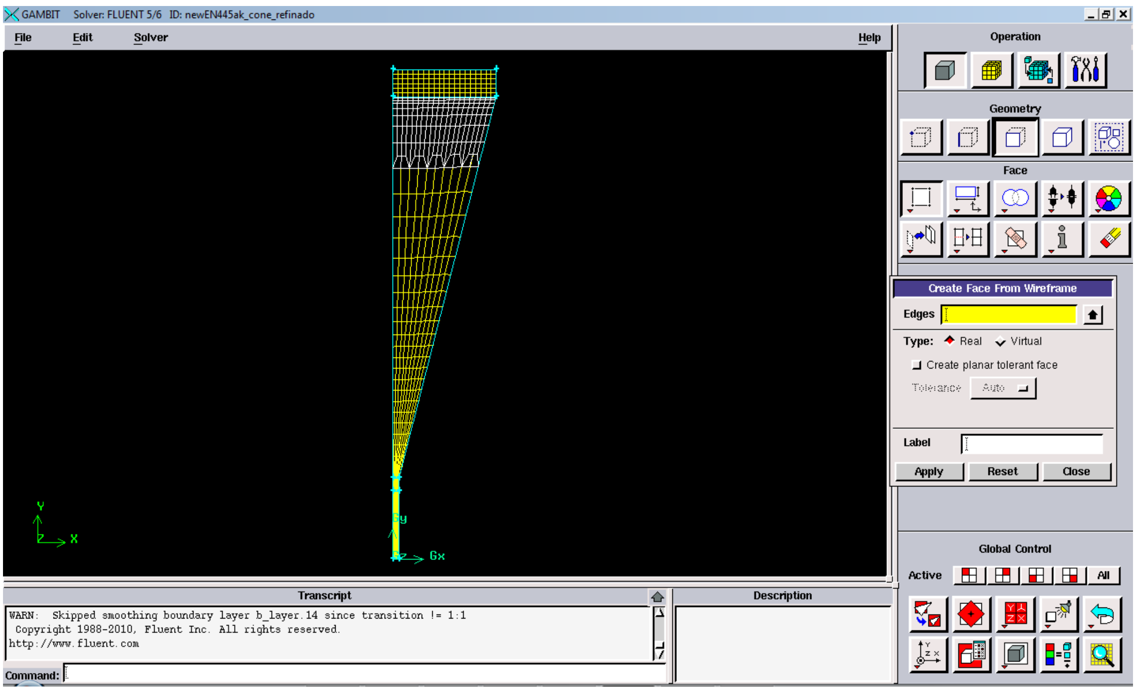

Figure 8.1: Marsh cone mesh generated with the software GAMBIT adopted in the numerical simulations.

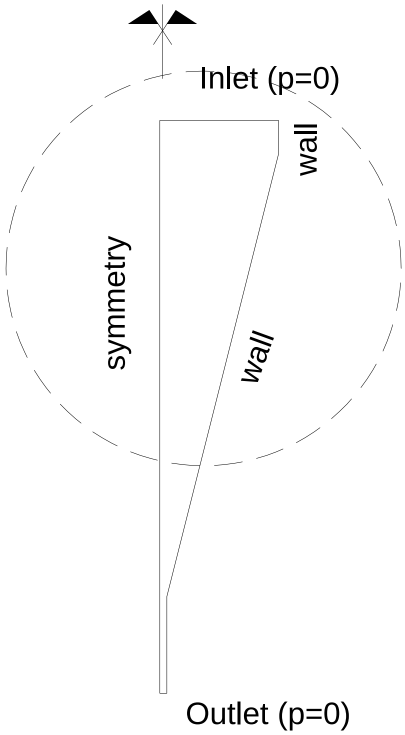

Figure 8.2: Marsh cone boundary conditions adopted.

The parameters of the mesh such as the number of nodes, of faces (edges in 2D) and of cells are also given in the following Table 8.2:

Table 8.2: Mesh parameters adopted.

Mesh parameters		
Number of nodes	Number of faces	Number of cells
2659	5178	2520

The rheological characteristics of the studied Bingham fluids are introduced into the numerical computation code assuming that the power-law index (*n*) is equal to 1 and the yielding viscosity is 10^6 Pa.s (very high value). According to (Nguyen *et al.*, 2006) the yielding viscosity should be sufficiently high to ensure that the numerical results are almost not changed. For that, the maximum average velocity of grout at the exit of Marsh cone (Fig.8.3) was compared with the same value for a model with different yielding viscosity input. The relative difference between a yielding viscosity equal to 10^6 Pa.s and equal to 10^5 Pa.s is only 0.65% (less than 1% error). Thus, 10^6 Pa.s was chosen for that parameter. Above this yielding viscosity value, it was observed that the numerical results are almost not changed. This means that in the Herschel-Buckley equation (Eqn. 6.1) *A* represents the plastic viscosity.

A sensitivity analysis was performed in order to determine the effect of "no-slip" parameter in Marsh cone walls, using FLUENT. This condition leaded to the best approximation of the test results. Thus, the boundary conditions of the walls were set to "no-slip".

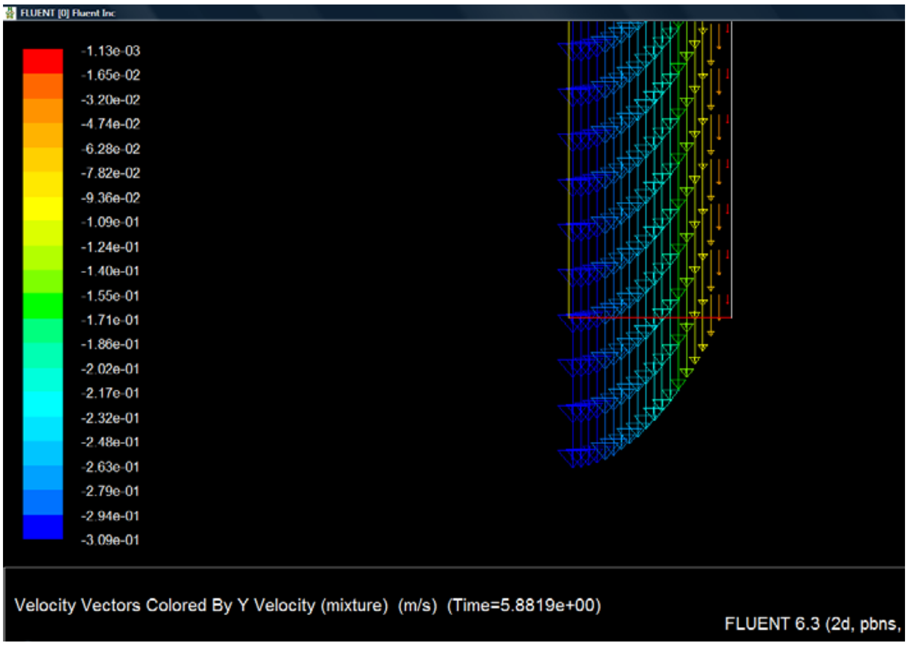

Figure 8.3: Maximum average velocity of grout at the exit of Marsh cone for yielding viscosity equal to 10^6 Pa.s.

In order to improve the convergence of the problem without changing the final results, it was assumed an initial velocity at the exit of the Marsh cone equal to 0.01 m/s.

According to (FLUENT, 2006), the time-dependent with the implicit interpolation scheme should be adopted if the shape of the liquid interface is important. Therefore, this scheme was the first adopted in the Marsh cone simulations.

Three different NHL5 based grouts (Table 8.1) were simulated using the previous assumptions. Results accuracy was improved by adjusting the time step size and the maximum iterations per time step. Because the computation time was too long, it was adopted the first order for the unsteady formulation integration instead of the second order. With this, data accuracy was kept and computation time reduced almost two times.
However, the numerical computation time is very long if an implicit VOF scheme is adopted, varying from a few hours to several days, depending on the flow velocity of each grout. Two

different PC were adopted: Intel ® Pentium® 4 (R) 3GHz 800 MHz FSB and a Intel ® processor Core 2 Duo T9300 2.5 GHz, 800 MHz FSB. With the processor Core 2 Duo, the numerical computation time for these grouts is 4 days approximately (for the standard cone) and 10 days for the improved Marsh cone shape (see 8.6). With the Pentium 4 no main differences were detected when comparing with the improved processor.

Due to the long computation time, an explicit interpolation scheme was also tested in Marsh cone simulations. This formulation can also be used for cases in which the flow calculation becomes unstable (FLUENT, 2006). With this new interpolation scheme, the computation time may becomes 50 times faster (or even higher) comparatively with the implicit scheme, without changing significantly the final results precision.

Since there is an intermediate layer in which both phases (air and grout) are present, it is impossible to obtain an ideal free surface which separates clearly the two phases. However, the thickness of this intermediate layer is small (from 2 to 3 cells) which ensures that the iso-surface of fluid (or air) volume fraction equal to 0.5 satisfactorily represents the free surface of the flow. Given that good results were obtained with this previous formulation, explicit scheme was adopted in the following tests.

8.6 Results and comments

The flow time obtained using numerical simulation to fill a vessel with 1litre of NHL5 based grout with w/b=0.60 is presented in Figure 8.4. The flow time obtained in the simulations was approximately the same if it was adopted an implicit or an explicit interpolation scheme.

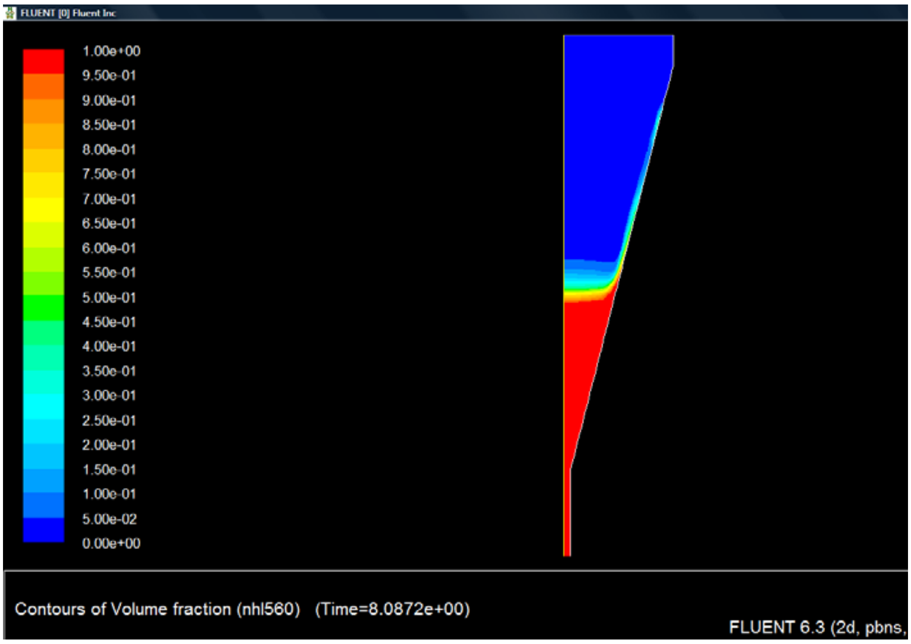

Figure 8.4: Flow time obtained using numerical simulation to fill a vessel with 1litre of NHL5 based grout with w/b=0.60.

Experimental values of flow time for the natural hydraulic lime grouts with different w/b ratio were determined according to EN445 and presented in Figure 7.7.

Figure 8.5 presents the comparison of the flow times given by the numerical simulation with those measured experimentally. It is noted that both types of values are very close only if the grout has w/b= 0.60. For higher water content the deviation is superior since it was obtained more flow diffusion for w/b=0.80 than to w/b=0.70.

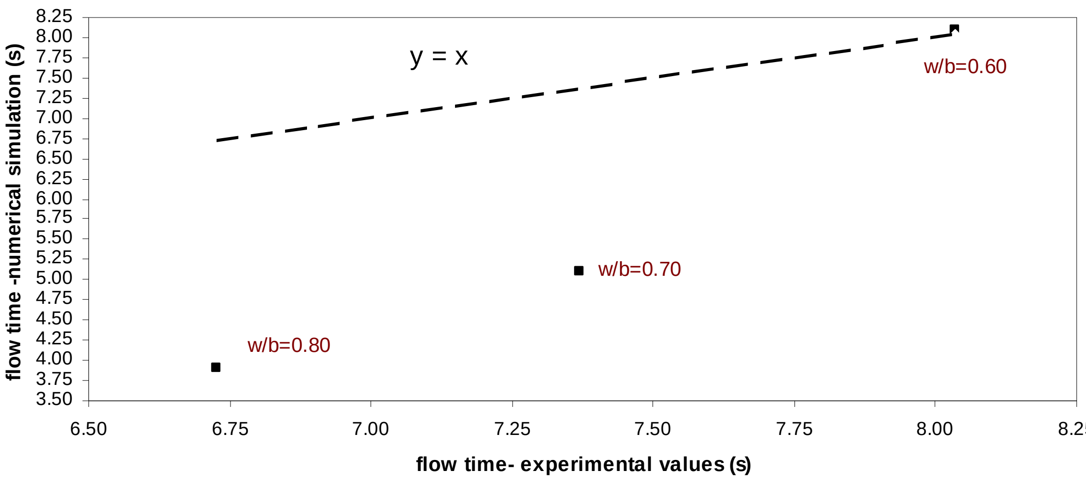

Figure 8.5: Comparison of the flow times for 1litre of grout given by the numerical simulation with the flow time measured experimentally.

This allows the conclusion that the numerical simulation of the flow of Bingham fluids through the Marsh cone is only validated satisfactorily for the lower w/b tested. The accuracy of those results was also checked by the adoption of a smaller time step size and even of a finer mesh to increase the precision.

To confirm if the type of flow was well selected, the Reynolds number was calculated (Table 8.3). It was observed that for all w/b tested the flow is laminar (the Reynolds number lower than 3000) which is in agreement with the definitions adopted in simulation.

Table 8.3: Reynolds number for each w/b ratio of NHL5 grout that flow though Marsh cone.

Marsh cone	
w/b	**Re**
0.60	111.2
0.70	214.4
0.80	339.2

The pressure drop in a fluid flowing through a long cylindrical pipe is given by the Poiseuille equation. The assumptions of the equation are that the flow is laminar viscous and incompressible and the flow is through a constant circular cross-section that is significantly longer than its diameter.

Concerning the cylindrical part of Marsh cone, if the distance between the entry and the end of the pipe is not enough, Poiseuille flow type will not occur and the grout flow will be turbulent for velocities and pipe diameters above a threshold. This will lead to larger pressure drops than would be expected according to that equation. The minimum distance to get this flow type is the length of establishment and depends on the Reynolds number and on the diameter of the pipe. The more the Reynolds number and the diameter is, the more distance is needed.

In the case of the Marsh cone, the Poiseuille flow occurs in the nozzle only when the Reynolds number is sufficiently small (lower than 10 approximately) which means that it will occur only for fluids of higher viscosity. For less consistent fluids, the velocity may be overestimated according to the Reynolds number because the assumption of Poiseuille flow type in the nozzle is not verified (Nguyen *et al.*, 2006).

According to Le Roy *et al.* (Le Roy *et al.*, 2004) the low viscosity grouts are responsible for the excessive debit in Marsh cone, which leads to model discrepancy. This phenomenon could explain the results in Fig. 8.5. Roussel *et al.* in (Roussel *et al.*, 2005) also states that for low viscosity fluids, the flow time value is not a meaningful measurement on rheological point of view. This can be corrected by choosing a narrower nozzle.

In practice it should be emphasised that the Marsh cone has to be used within its application domain to reach its maximum efficiency as a control tool (Le Roy *et al.*, 2004). A larger number of tests are necessary to ensure that the numerical simulation approach is verified in a wide range of rheological characteristics of grouts.

8.7 Marsh cone improvement

According to the previous results presented in Chapter 7, the Marsh cone test seems to be inadequate for grout design for injection purpose at least for the grain size distribution type “2-4”mm of the porous media, or even smaller.

This conclusion suggests the use of an improvement of the Marsh cone shape for test grout flow time for masonry injection purpose. Thus, a new Marsh cone geometry was constructed and tested experimentally, with a thinner cylinder at the end (with radius equal to 3mm and height equal to 54 mm) adapted to the EN445 cone. A new mesh for numerical simulation was generated using the same procedures previously described. Fig. 8.6 presents the “new” Marsh cone shape used in numerical simulations as well as the initials and boundary conditions of the flow. The mesh includes the area from the cone until the vessel.

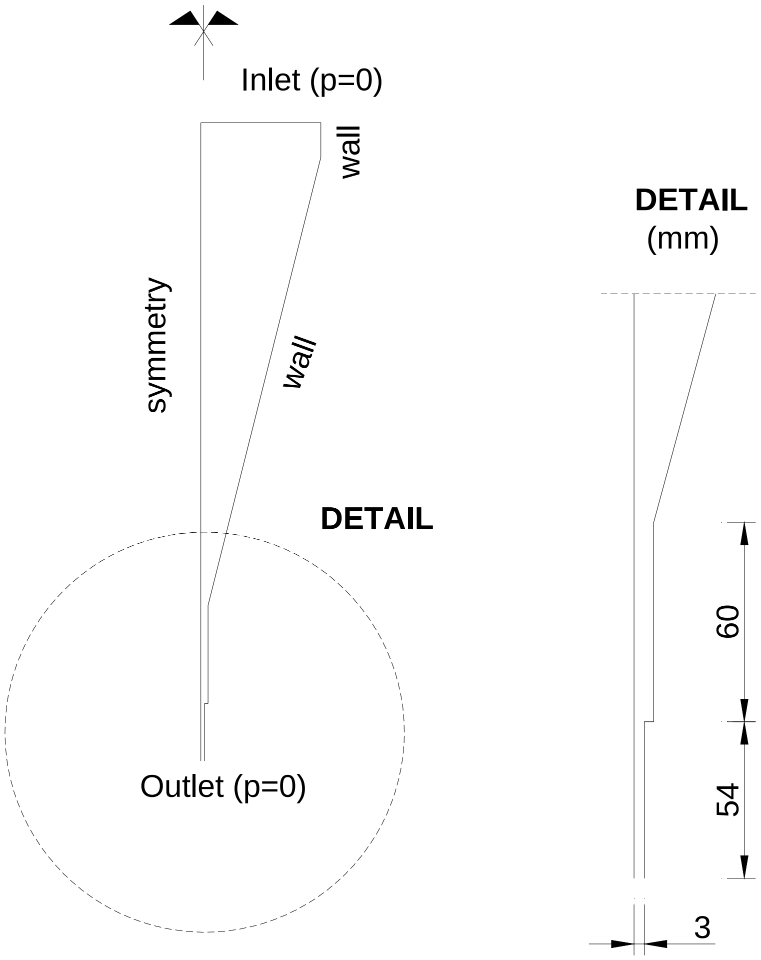

Figure 8.6: “New” Marsh cone shape used in numerical simulations as well as the boundary conditions.

The flow time for a NHL5 grout with w/b=0.70 given by the numerical simulation is presented in Fig. 8.7.

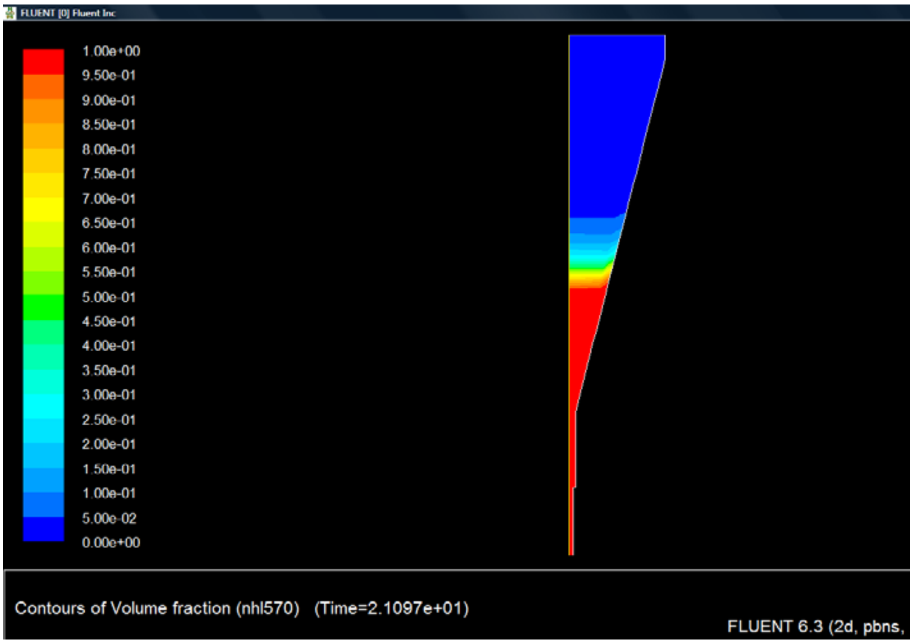

Figure 8.7: Flow time obtained using numerical simulation to fill a vessel with 1litre of NHL5 based grout with w/b=0.70, for the new shape for Marsh cone.

The comparison of the flow times given by the numerical simulation with the flow time measured experimentally is presented in Fig. 8.8.

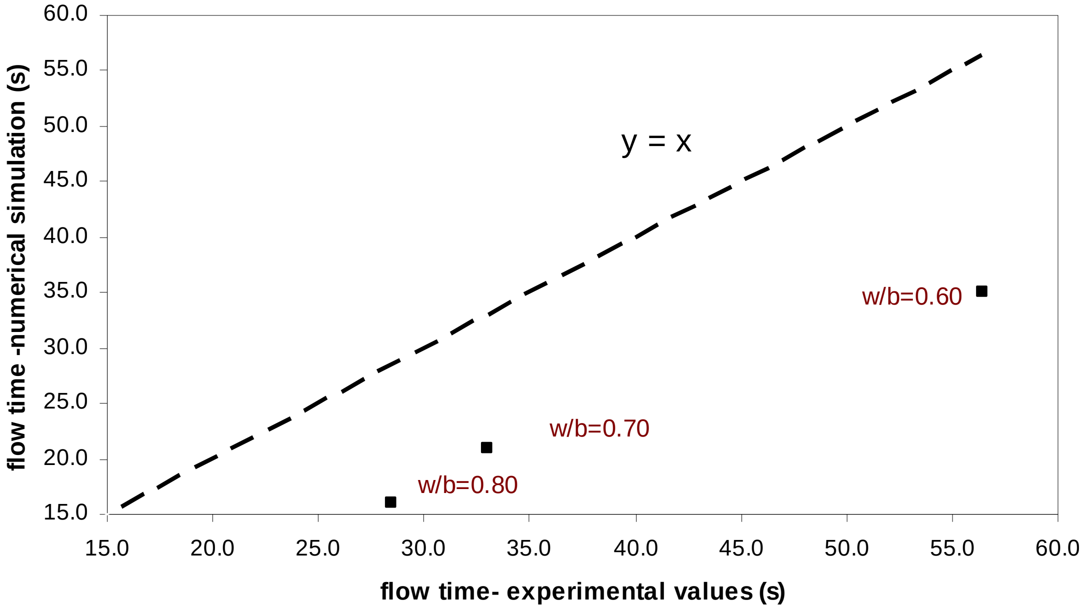

Figure 8.8: Comparison of the flow times for 1litre of grout given by the numerical simulation with the flow time measured experimentally, for the new shape for Marsh cone.

It is noted that the calculated flow time and the flow time obtained by numerical simulation are closer only if the grout has w/b= 0.70. Thus, for the thinner cylinder tested, the precision was increased for a water range near to 0.70. For less or higher water content the accuracy is smaller.

Like for the EN445 Marsh cone, again it could be state that for less viscosity fluids (higher water content), the velocity may be overestimated according to the Reynolds number because the assumption of Poiseuille flow type in the nozzle is not verified again. It means that for w/b higher than 0.70 the ratio between radius and length of the cylinder should be smaller to increase the precision for that water content.

On the other hand, the lower water content of the grout is, the more the difference between experimental and numerical simulation times. The lower water content is associated to a lower flow velocity and the lower the experimental flow velocity of grout is, the more the time step must be decreased to ensure the solution convergence and stability while the more the flow time of 1 litre of grout increases (Nguyen *et al.*, 2006). This previous situation was also taken into account in these numerical simulations.

Thus, it seems that this modified Marsh cone is appropriated for a certain maximum and minimum water content. Concerning the minimum, it seems to be somewhere near w/b=0.60, below that the cone "does not work". In fact, this previous water content (flow time equal to 8s) seems to be well represented by the EN445 Marsh cone (see Fig. 8.5).

Since the flow time of the grout with w/b=0.60 is high, the difference between the experimental and the numerical flow time for this water content could be explained by the beginning of thixotropic effect, because the material builds up an internal structure which is associated to a viscosity increase with time. Since the numerical simulation did not take into account this phenomenon, it takes less time than the experimental situation. So, it means that there is a domain in each "type" of Marsh cone where thixotropic effect does not need to be taken into account in experimental situation neither in numerical simulation. Marsh cone has to be used within its application domain to reach its maximum efficiency as a control tool.

In terms of "absolute minimum" of flow time in a certain Marsh cone shape, it is related to the yield stress of the grout. If the grout has a yield stress, the flow may not occur because the pressure gradient created by the fluid weight above the nozzle may not be enough for the shear stress to overcome the yield stress (Roussel *et al.*, 2005). The flow only occurs if (Eqn 8.2):

$$\frac{2\tau_0}{Ar} \leq 1 \tag{8.2}$$

Where τ_0 is the grout yield stress, A is the pressure gradient motor of the flow and r is the radius of the cylinder.

Flow does not occur if the rate of flow is equal to zero. Thus, the pressures gradient is (Eqn 8.3):

$$A = \rho g \frac{H}{h} + \frac{8\tau_0}{3h\tan(\alpha)} \ln\left(\frac{r}{r + H\tan(\alpha)}\right) \tag{8.3}$$

Where H is the height of the cone filled by grout (above cylinder height), h is the cylinder height, α is the opening angle of the cone and ρ is the grout density.

For each tested grout the criterion values were calculated using Eqn 8.2 and 8.3 for the EN445 shape and for the modified Marsh cone shape (Table 8.4):

Table 8.4: Verification of the flow criterion for the tested grouts.

	Stop flow criterion	
w/b	**EN445**	**Modified shape**
0.60	0.021<1	0.065<1
0.70	0.009<1	0.028<1
0.80	0.003<1	0.01<1

In can be observed that for all the tested grouts the flow occurs of the two shapes tested, at least until the final height to fill the vessel with 1 litre of grout is achieved.

No references concerning simulation of grout flow with higher w/b ratio (higher flow time) were found. (Nguyen *et al.*, 2006) have numerically modelled the flow of five cement grouts with low w/b: 0.60, 0.55, 0.50, 0.45, 0.42 through the Marsh cone by pursuing the numerical calculation until 1 litre of grout flowing out. The grout rheological behaviour was the Herschel–Bulkley and it was concluded that the numerical simulation of the flow of those fluids through the Marsh cone (according to EN445) is good.

However, shape modification represents a huge difference in terms of flow time: for the NHL5 based grouts developed and injected in a porous media as explained in Chapter 7, the experimental results obtained allow the same conclusion concerning the w/b ratio after what no significant changes are observed ($w/b \geq 0.70$) (Fig. 8.9).

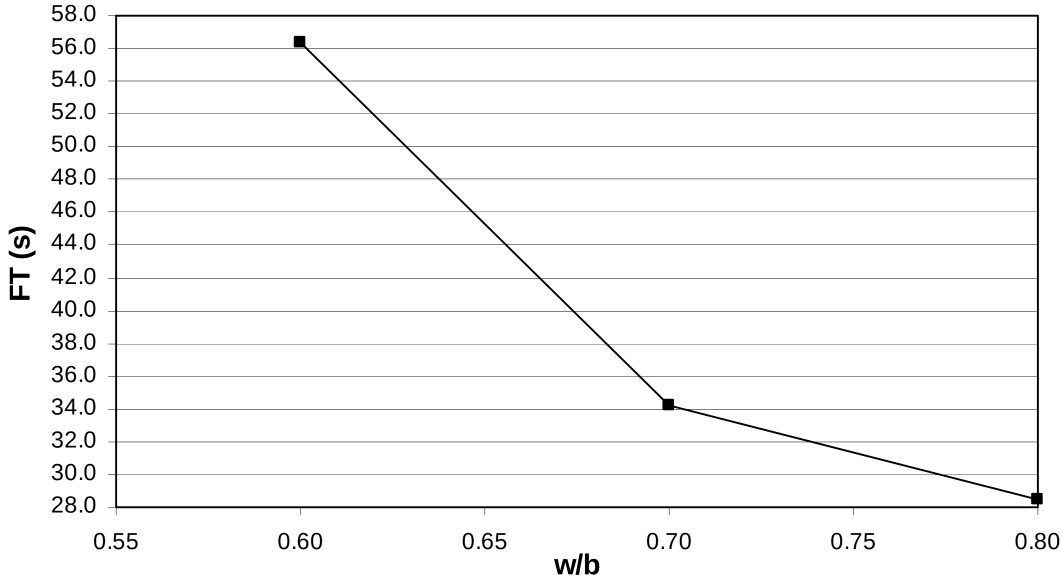

Figure 8.9: Flow time for 0 minutes after grout preparation for different w/b tested in the new Marsh cone device.

The rheological results in terms of yield stress and plastic viscosity for NHL5 based grouts with w/b= 0.60; 0.70 and 0.80, used in the numerical simulation are presented in Fig. 8.10. The rheological parameters were calculated from the upward curve (going from a lower degree of dispersion to a higher one), using the procedure described in 7.4.4 of Chapter 7.

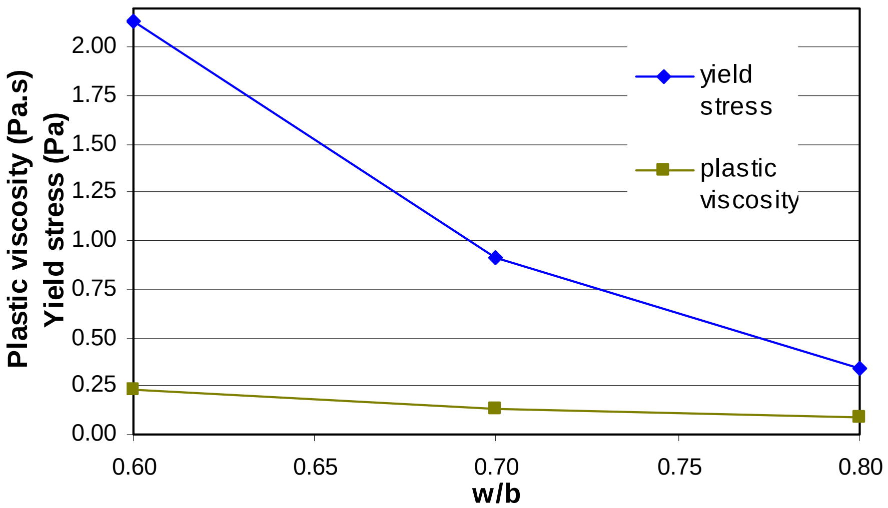

Figure 8.10: Rheological results in terms of yield stress and plastic viscosity for NHL5 based grouts with w/b= 0.60; 0.70 and 0.80 used in the numerical simulation.

The analysis of rheological parameters shows that there is a considerable decrease of yield until w/b reaches 70%, after which that parameter does not change that much. The comparison between the slope of yield stress and the "new" flow time for different water content (Fig. 8.9 and 8.10) shows that this parameter seems to be the one that has more influence in flow time results.

Again, those results prove the influence and the importance of rheological properties in grout design and injectability, as confirmed by the injection tests.

8.8 Main conclusions

The present study aims at relating the rheological parameters of the tested grouts to their flow time through the Marsh cone which characterizes in a practical way the fluidity of grouts. A comparison between the numerical simulation results of Marsh cone was made with the experimental ones. The main conclusions are:

- Explicit interpolation scheme seems to give good results in the numerical simulation tested.
- The calculated flow time and the flow time obtained by numerical simulation of Marsh cone according to EN445 are very close only if the grout as w/b= 0.60. For higher water content the deviation is superior. Numerical simulation of the flow of Bingham fluids through the Marsh cone is validated satisfactorily only for the lower w/b tested probably meaning that other rheological behaviours should be defined.
- Grout flow time using the device according to EN445 does not change much between $0.60 \leq w/b \leq 0.80$. It means that, from this point of view, there are no benefits if w/b is increased beyond 0.60 which is not true if injectability analyses of those grouts are made (Chapter 7).
- Marsh cone shape modification leads to flow time results with the same slope as the ones of injectability.
- The new shape is an evidence of the importance of grout rheological behaviour in decisions concerning grout composition.
- It seems that each Marsh cone is appropriated for a certain maximum and minimum water content, which is related to a minimum and maximum grout flow time.
- Flow time is more related with yield stress than with grout plastic viscosity.

- More tests should be made to get a good agreement between the numerical results and experimental values, in order to allow us to use those results to predict flow time in Marsh cone device. Herschel–Bulkley model should also be applied.
- Since this commercial software is not an open source, it is not possible to know how is the numerical methodology and code used.
- No main differences were detected if an implicit VOF scheme is adopted in the numerical computation time when using a Intel ® Pentium® 4 (R) 3GHz 800 MHz FSB or a Intel ® processor Core 2 Duo T9300 2.5 GHz, 800 MHz FSB. It means that FLUENT 6.3.26 is not optimized for the multi-core processors. Recently, ANSYS, Inc. has developed a new version of that CFD program, ANSYS® 12.0. The high performance computing achievements in the recent release include optimized parallel computing performance on multi-core processors, expanded support for large simulations, scaling breakthroughs and support for parallel file systems (ANSYS, Inc.). ANSYS 12.0 technology incorporates optimization for the latest multi-core processors and benefits greatly from recent improvements in processor architecture, resulting in highly efficient use of parallel processing to reduce the numerical computation time. This new technology should be adopted in future research works in order to optimize product development processes and take faster decisions.

8.9 References

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Chapter 9. Conclusions and recommendations

9.1 Conclusions and recommendations

A methodology regarding grout design for masonry injection has been developed and it was shown that grouts can be optimized.

The laboratory tests in this research have basically been performed in the following stages:

- Definition of an optimal mixture procedure for a best grout performance based on the necessary requirements for injection purpose (Chapter 4).
- Determination of an optimal grout composition for injectability, strength and durability point of view, by using the Taguchi method (Chapter 5). This method also enables the reduction of scatter in grout properties leading to its improvement.
- The use of rheology during grout mix design for masonry injection purpose. Steady state and transient state material's characterization enabled to get that information and to make a decision about it (Chapter 5 and 6).
- Grouts characterization based on maximum resisting time, rate of flocculation and analysis of the hydraulic lime grout behaviour tested at different shear rates, was performed using a shear thinning model and assuming that the structure is shear and time dependent. The goal is to use this methodology during mix proportioning and design for masonry injection purpose (Chapter 6).
- Besides the study of the best fresh and hardened grout properties, the study of the interaction between the optimized grout and masonry is of major importance. To improve the knowledge about the physical mechanisms that take place during injection and determine the penetration of the grout in the masonry, reproducible masonry samples of cylindrical shape were used. Injection tests were performed in those cylindrical samples. The study has envisaged to define an adequate composition for grouts to be used for the strengthening of walls (Chapter 7).
- An attempt to get an injectability curve for each grout tested as a function of percentage of total mass that passes through the fine grained soil, the porous media porosity and taking into account the void diameter average was also made (Chapter 7).
- To get a relation of the rheological parameters of the tested grouts to their flow time through the Marsh cone which characterizes in a practical way the fluidity of grouts on field.

- A comparison between the numerical simulation and experimental results of Marsh cone was made. The goal was to get a good agreement between the numerical results and experimental values, in order to confirm the validity of the numerical approach and allow us to use those numerical results obtained for Marsh cone to predict grout flow time (Chapter 8).

The definition of an optimal mixture procedure is crucial for a best grout performance. It was observed that the period of mixing is important since a greater mixing time leads to a better wetting. Stability tests show that there are different results when using different grout mixing procedures. Some of them lead to more free water than others, and this means that less stable grout will be obtained, which is not good.

Besides that, other parameters, such as grout rheological behaviour and water retention capacity, change with the selected procedure. All these facts suggest that the addiction of water to binder (and not the opposite) produces an inefficient mixture. Since this capacity is essential for grout flow inside masonry, again it is possible to highlight the need to get the optimal way to mix grout.

According to grout injection requirements, an attempt was made to achieve an optimal grout composition by using the Taguchi method. The use of the Taguchi method represents a good option and allowed a considerable reduction of the number of experiments, when compared to the full factorial, therefore optimising the controllable inputs and measurable outputs. It enables also the study of the influence of each factor (variable) in final result, allowing grout composition optimization for injection purpose.

For the range of grout compositions tested, the optimum grout obtained was the one with w/b=0.70 together with a fly ash content= 15%, not only in terms of fresh state but also hardened properties results.

The results show that fly ash is capable of substantially changing the grout rheological parameters, while the effect of temperature is negligible for the time independent properties tested (such as yield stress, plastic viscosity, consistency and power-law index). It is then possible to control those parameters in order to get an improvement of injection capacity but also in terms of strength and durability since there is also a reduction of the higher size category of grout voids diameter and reduce permeability, which represent a strength improvement (as it was previously confirmed).

The developed research also aims at contributing to better understand the flow behaviour that natural hydraulic lime-fly ash grouts presents under different temperatures, taking into account not only the steady state but also the transient one. Thus, the goal of rheological measurement designed in this work (Chapter 6) was to establish a determination of the thixotropy of each grout and hence compare how structured are the mixes in relation to their variables of preparation and test conditions. An attempt to find the temperature limit (T_{limit}) that isolates thixotropy effect from hydration reactions in a grout was made. For NHL5 based grout T_{limit} =20ºC and for NHL5+15% fly ash based grout T_{limit} =15ºC.

It was shown that the replacement of NHL5 binder by 15% of fly ash leads to yield stress and plastic viscosity decrease. However it was also detected a thixotropy decrease.

Grouts characterization based on maximum resisting time, rate of flocculation and analysis of the hydraulic lime grout behaviour tested at different shear rates, was performed using a shear thinning model and assuming that the structure is shear and time dependent. The goal is to use this methodology during mix proportioning and design for masonry injection purpose, for raw materials evaluation or even in quality control *in situ*.

However, the lack of information about which is the best solution for grout design, as a function of the porous media to be injected, enhances the importance of a detailed research on the subject. Thus, to improve the knowledge about the physical mechanisms that take place during injection and that determine the penetration of the grout in the masonry, reproducible masonry samples of cylindrical shape were used.

It was observed that during cylinders injection only grouts with $w/b \geq 0.60$ may be injected in a porous media type “2-4”mm. There is also an expressive decrease of injection time for a grout with $w/b \geq 0.70$, mainly for 1.0 bar and 1.5 bar. This means that the grout penetration capacity is optimized for that water content. In fact, the injection time value became almost constant for $w/b \geq 0.70$, even for an increasing injection pressure. The analyses of the rheological behaviour, especially plastic viscosity of grouts lead to the same conclusion: it became constant only for that w/b range (and not for $w/b \geq 0.60$) - which proves the influence and the importance of these properties in grout design and injectability, as confirmed by the injection tests.

An attempt to get injectability curves for NHL5 and NHL5+15% fly ash based grout was made (see 7.5.5). Some researchers, such as Binda, have studied masonry walls of buildings and tried to calculate its voids. The survey of the internal sections allowed to define some important parameters like the percentage distribution of stones, mortar, voids which allows to make comparisons between the different regions. If it possible to obtain a grain size distribution from the interior of a masonry to be injected and put similar porous media in a device like 7.4.5, then an estimative of what is the grout injection capacity can be made.

In terms of binder composition, fly ash tended to increase tensile strength, enabling a good bonding of the grout to the masonry and improving load bearing capacity of the structure. Probably, fly ash reduces porosity and increases densification of interfacial bond between aggregate and grout, explaining the previous results of splitting test (7.5.6). SEM results of the tested samples would help in these confirmations.

Analysis regarding an higher grain size distribution of the porous media (such as type “4-10”mm) indicate that the grain size distribution of the porous media to be injected has an important influence on the type of analysis that should be made in grout design for consolidation purpose: Marsh cone should be modified and adopted for a certain grain size range of masonry in order to become an effective and quick test tool to apply on field for grout design.

Marsh cone shape modification represents a considerable difference in terms of flow time (see Chapter 8). Those flow time results present the same slope as the ones of injectability. The new shape is an evidence of the importance of grout rheological behaviour in decisions concerning grout composition. It could be said that flow time is more related with yield stress than with grout plastic viscosity.

A comparison between the numerical simulation and experimental results of grout flow time in a Marsh cone was made. Numerical simulation of the flow of Bingham fluids through the Marsh cone is validated satisfactorily only for the lower w/b tested (0.60). The goal was to get a good agreement between the numerical results and experimental values, in order to confirm the validity of the numerical approach and allow us to use those numerical results obtained for

Marsh cone to predict grout flow time. This will enable to take decisions, predict problems and correct grout compositions before application on field.

This research has increased the understanding of how is the behaviour of some grout compositions during the fresh and hardened state. However, the measurements have been performed in a laboratory environment and it means that some outside parameters will probably have an impact in some results. As the proposals for future research are described in Chapter 10, the material properties investigated in this research work must be incorporated in a more comprehensive study for grout development concerning several specific applications. The methodology presented in this thesis will provide part of the tools necessary for that purpose.

Based on the references recommendations and author experience during the research work, Table 9.1 presents some rheological recommendations for grout design.

Table 9.1: Rheological recommendations for grout design.

Requirement	Situation
Grout with < plastic viscosity	When grout is forced to flow with high speed between small voids (means high shear rate).
Grout with > plastic viscosity	In situations where there are large voids to fill and flow should be at low speed (the shear rate will be small) like fiber reinforced concrete columns by jacketing. The fiber will stay inside the concrete for > plastic viscosity.
Grout with < yield stress	For self-levelling situation. For grout injection inside porous media with void diameter (D) yield stress should not satisfy Eqn. 7.5.
Grout with > yield stress	To minimize grout stability problems.
Grout with < consistency	To reduce grout apparent viscosity, meaning that it will increase less for a shear rate increase.
Grout with > consistency	To increase grout apparent viscosity.
Grout with power-law index < 1	When a shear thinning effect is expected. If flow velocity decreases during grout injection it leads to a viscosity increase which will lead to stop flow. Pressure of injection pump should be carefully controlled, namely for really low pressure (where the viscosity variation is higher).
Grout with power-law index = 1	Grout viscosity will be constant for a shear rate variation (Newtonian behaviour). Better in general situations, especially for pump systems less developed.
Grout with power-law index > 1	When a shear thickening effect is expected (viscosity increase for an increase of flow velocity).
Grout with $> A_{thix}$	If grout injection is slowly enough or if it is at rest, it builds up an internal structure and has the ability to withstand the load from grout injected above it. Because the material builds up an internal structure, the lateral stress in masonry decreases quickly in the part of the injected grout that is at rest inside the porous media, avoiding structural damage.
Grout with $< A_{thix}$	If grout flocculates too much faster, then, the apparent yield stress will increase above a critical value and the two adjacent grout layers do not mix at all. This probably will create a weak interface in the final structure. Thus, a grout with less A_{thix} should be selected.
Maximum shear rate during grout mixing	$300 s^{-1}$

Chapter 10. Future research

10.1 General aspects

Further research in some areas is necessary to improve grout design concerning several specific applications. From an injection point of view, additional characterization of the flow and injection processes are needed, due to the complex composition of grouts and the interaction of flows and flow geometries which may evolve in time. Some suggestions will be presented in the following topics.

10.2 Laboratory and field studies

Basically, a good grout is the one that satisfies certain requirements according to the specific application. Thus, the goal is to design a homogeneous grout that is easily placed at the lowest possible cost. Rheology is a major tool in this context. It can be used during mix proportioning or mix design, for raw materials evaluation or in quality control at building site.

However, in rheometry, many disturbing effects may arise. They often reflect the influence of the microstructure. For instance, for a particle suspension, as is the case of grout, mortar or concrete, sedimentation and migration of particles can significantly change the stress distribution and thus the measured torque. Likewise, for concentrated grouts, a fracture inside the sheared sample may sometimes be observed, usually resulting from a localization of shear within a thin layer. Other disturbing effects are experimental problems pertaining to the rheometer type. For example, when using a rotational viscometer with a smooth metallic shearing surface, wall slip can occur and disturbing measurements may appear.

A great deal of effort has been spent to obtain accurate and repeatable data on the rheological parameters but they are often inconsistent depending on the technique or instrument used. Thus, rheology has not been so much applied as it should and the evaluation of workability has been dominated by several empirical and simple test methods, as is the case of the Marsh cone.

Nevertheless, it is possible to detect some general trends in terms of rheological behaviour, knowing for example what is the effect of the addition of water, fly ash, silica fume, superplasticizers, among others, in grout yield stress or plastic viscosity. The effect of each constituent is now becoming known, enabling material optimization. However, more studies should be conducted in order to get detailed and correct information, since the effect of grout composition becomes different if the suspension is more or less diluted, if there are changes in particle size and form (the finer the cement is, the more colloidal forces will influence its movement), if binder type is changed, or if others changes are induced.

Injection grouts should be well conceived, taking into account the optimization of their performance. Whenever simple binder and water formulations prove inadequate, which happens frequently, the use of superplasticizers, mainly organics, or silica fume (inorganic materials) to improve rheology and stability of the grouts, thus optimizing the injection characteristics, may be considered. Taking into account that the need for conservation of the masonries is crucial, durability concerns should also be taken into account.

The addition of superplasticizers results in an improved dispersion of hydraulic lime or cement grouts and it means that flocculation is reduced or even prevented. The rheological properties are improved (at least yield stress is reduced) and flow occurs more easily. The addition of colloidal silica into grout composition increases its thixotropic behaviour, which has the ability to form loose three-dimensional structures. This phenomenon should be studied and controlled in order to take an advantage on that in grouts development (as it was mentioned in previous chapters).

The lack of information about the influence of these additives in the rheological behaviour, particularly in what concerns stability, strength, durability and modes of action in grouts, emphasizes the importance of the proposed detailed research work. Thus, a contribution to the improvement of knowledge affecting both the scientific community and industry should be made. The Department of Civil Engineering of UNL is now working on that subject, through a financed research project: Advanced Grouts for Masonry Consolidation (project reference: PTDC/ECM/104376/2008).

The determination of injectability curves for several grouts and for different porous media will help in the choice of the most suitable grout composition. More efforts should be made for that purpose, taking also into account a survey concerning characterization of Portuguese masonries. The results aim at enabling the planning and design of more efficient grout for masonry injection.

Numerical models for grout behaviour prediction should also be developed and a particular attention must be given to empirical and simple test methods applied on field. They have proved throughout the years to be able to classify different materials in terms of their ability to be applied in each situation: Marsh cone for fluidity or the slump test in the evaluation of concrete casting ability. The good agreement between the numerical results and experimental values will confirm the validity of numerical approach and will allows the use of those numerical results to predict material behaviour on field.

The use of computational models for the evaluation of flow in porous media is foreseen, aiming at the definition of numeric evaluations of the injection capacity and global performance of grouts, providing also information concerning the best grout solutions (in terms of optimum flow behaviour in each situation and in terms of best material composition) and avoiding problematic situations that could occur on field if no previous studies were made.