THE CONSEQUENCES OF THE DEWATERING OF FRESHLY-MIXED WET MORTARS BY THE CAPILLARY SUCTION OF BRICK MASONRY

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ABSTRACT

Name of the University: The University of Manchester
Submitted by: Nidhal Al-Defai
Degree Title: Doctor of philosophy
Thesis Title: The consequences of the dewatering of freshly-mixed wet mortars by the capillary suction of brick masonry
Date: 30-08-2013

The main water transport properties of clay brick are critically examined in respect of suction and water content. An experimental investigation is carried out to compare the sorptivity and vacuum saturation porosity with suction and “porosity” tests in the standards.

The water retaining ability (desorptivity) of freshly mixed hydraulic lime and cement mortars is examined and the effect of hydraulicity, pozzolanic and non-pozzolanic additive materials, binder particle size and the chemistry of mix water on the water retentivity of these mortars are investigated.

The inter-relationship of substrate (brick) suction and desorptivity of freshly mixed wet lime and cement mortar are investigated. It is shown that the initial setting time of dewatered freshly-mixed mortars is reduced by a factor of up to 80% and the final setting time is reduced by a factor of 60%. The extent of this reduction depends on hydraulicity. For the cured mortars, following dewatering in the wet state, the compressive and flexural strengths are increased by about 40% for cement mortar and by more than 3 times for lime mortar. The sorptivity of hardened cement and lime mortars is reduced by 80%. These results have implications for the British and European standards where mortars are cast in impermeable steel moulds in which dewatering cannot occur prior to setting.

The accuracy of the methodology of the American Petroleum Institute (API) pressure cell technique for testing the water retaining ability of fresh mortars was critically examined. An experimental investigation was carried out in two parts, first by changing the controlled variables of the experimental set up. Second the consequences for the results obtained were evaluated.

Experimental verification is undertaken of the fundamental Sharp Front equation \( S = (2K\Psi f)^{1/2} \) which describes the inter-relationship of capillary pressure, sorptivity, porosity and hydraulic conductivity.
DECLARATION

I hereby declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.
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NOTATIONS

Some symbols such as constants of integration are defined within the text and are not repeated in this list.

- $C_2S$: Dicalcium silicate.
- $C_3S$: Tricalcium silicate.
- CEM II: Portland-limestone cement type 2.
- CL90: Calcium lime with at least 90% calcium hydroxide.
- GGBS: Ground granulated blast furnace slag.
- GBT: Gas breakthrough time.
- GP: Golden Purple brick.
- LB: Leicester Buff brick.
- NHL: Natural hydraulic limes.
- NHL5, NHL3.5, NHL2: The designator number after NHL is the minimum expected 28 days compressive strength in MPa.
- NHL5-Z: The designator Z is an addition of suitable pozzolanic or hydraulic materials, up to 20% by mass.
- SEM: Scanning electron microscopy.
- SF: Sharp Front theory.
- $A$: Transfer sorptivity ($mm/min^{1/2}$).
- $A_s$: Cross sectional area of the sample ($mm^2$).
- $A_c$: Cross sectional area of the water column ($mm^2$).
- $D$: Hydraulic diffusivity ($mm^2/sec$).
- $f$: Porosity (dimensionless).
- $f'$: Satiation porosity.
- $f''$: Satiation porosity under vacuum.
- $h$: Hydraulic head (mm).
- $h_t$: Tension head (mm).
- $i$: Cumulative volume of water per unite area (mm).
- $I$: Initial rate of absorption (Kg/m$^2$ min).
- $k$: Saturated Permeability (mm/min).
- $K$: Hydraulic conductivity (mm/min).
- $K_c$: Hydraulic conductivity of the filter cake (mm/min).
<table>
<thead>
<tr>
<th>Notations</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L$</td>
<td>Length of sample (mm).</td>
</tr>
<tr>
<td>$L_c$</td>
<td>Length of filter cake (mm).</td>
</tr>
<tr>
<td>$L_w$</td>
<td>Length of the wetted zone (mm).</td>
</tr>
<tr>
<td>$m_d$</td>
<td>Mass of dry sample (g).</td>
</tr>
<tr>
<td>$m_w$</td>
<td>Mass of wet sample (g).</td>
</tr>
<tr>
<td>$P$</td>
<td>Pressure (MPa).</td>
</tr>
<tr>
<td>$\Delta P$</td>
<td>Pressure difference (MPa).</td>
</tr>
<tr>
<td>$Q$</td>
<td>Quantity of water flowing per unit time (mm$^3$/min).</td>
</tr>
<tr>
<td>$R$</td>
<td>Desorptivity (mm/ min$^{1/2}$).</td>
</tr>
<tr>
<td>$R_f$</td>
<td>Flexural strength (MPa).</td>
</tr>
<tr>
<td>$R_c$</td>
<td>Compressive strength (MPa).</td>
</tr>
<tr>
<td>$S$</td>
<td>Sorptivity (mm/ min$^{1/2}$).</td>
</tr>
<tr>
<td>$S_{vis}$</td>
<td>Visual sorptivity (mm/ min$^{1/2}$).</td>
</tr>
<tr>
<td>$S_{vac}$</td>
<td>Vacuum sorptivity (mm/ min$^{1/2}$).</td>
</tr>
<tr>
<td>$S_r$</td>
<td>Repeatability standard deviation.</td>
</tr>
<tr>
<td>$S_R$</td>
<td>Reproducibility standard deviation.</td>
</tr>
<tr>
<td>$t$</td>
<td>Time (min).</td>
</tr>
<tr>
<td>$t_{dw}$</td>
<td>Time to dewater freshly- mix mortar (min).</td>
</tr>
<tr>
<td>$u$</td>
<td>Darcy velocity (mm/min).</td>
</tr>
<tr>
<td>$x$</td>
<td>Wet front advancing length (mm).</td>
</tr>
<tr>
<td>$z$</td>
<td>Gravitational head (mm).</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>Water: solids volume fractions in the fresh and dewatered states of the mortar.</td>
</tr>
<tr>
<td>$\theta$</td>
<td>Volume fraction water content (dimensionless).</td>
</tr>
<tr>
<td>$\theta_i$</td>
<td>Initial volume fraction of water in the fresh mix.</td>
</tr>
<tr>
<td>$\theta_c$</td>
<td>Volume fraction of water in the filter cake.</td>
</tr>
<tr>
<td>$\theta_w$</td>
<td>Volume fraction of water in the wetted zone.</td>
</tr>
<tr>
<td>$\phi$</td>
<td>Volume fraction solid content (dimensionless).</td>
</tr>
<tr>
<td>$\phi_i$</td>
<td>Initial volume fraction of solids in the fresh mix.</td>
</tr>
<tr>
<td>$\phi_c$</td>
<td>Volume fraction of solids in the filter cake.</td>
</tr>
<tr>
<td>$\psi$</td>
<td>Capillary pressure (mm) (accepted terminology instead of head).</td>
</tr>
<tr>
<td>$\psi_0$</td>
<td>Capillary pressure at the inflow faces (mm).</td>
</tr>
<tr>
<td>$\psi_1$</td>
<td>Capillary pressure at the wet front (mm).</td>
</tr>
</tbody>
</table>
CHAPTER 1  INTRODUCTION

1.1 OVERVIEW

The successful use of building materials requires an understanding of their properties as they affect the building at all stages of its lifetime. Masonry remains a basic part of most buildings. Even steel structures and timber frame buildings often have a masonry cladding. Hence it is essential to understand their values and possible problems. The greater part of masonry walling is produced from construction units bonded by a mortar. Clay bricks continue to be the most important building units, joining excellent durability (when selected and used correctly) with lasting aesthetic properties in the case of facing bricks.

Mortars may be defined as mixtures of a binder such as lime or cement, sand and water. Any cementitious binder used for mortars should fulfil many requirements. In the fresh state mortars should be workable, yet cohesive so that a good bond can be achieved with the masonry units. In the freshly-mixed state, the mortar is desired to have good water retention ability to resist the suction forces exerted by the porous substrates such as bricks or blocks and retain sufficient water for chemical hydration to continue. In the set and hardened state, mortars should resist freezing and thawing or other forms of environmental attack; they should resist penetration of water; and they should contribute to the visual appearance of the wall. Mortar should impart sufficient strength to the masonry structure, although the wall as a whole is about five times stronger than the mortar in view of the fact that the mortar is applied in a very thin layer (10-20 mm) [1].

In building construction there are a variety of mortars which are prepared from different types of binders and used for jointing and rendering purposes. The most commonly used mortars are lime, cement and composite mortars. Hydrated lime, all the range of hydraulic limes and Portland cements may be considered as a group of related materials which have a wide range of properties in both the soft and hardened states.

Hydrated lime (air lime) is made by heating relatively pure limestone containing more than 95% calcium carbonate to about 1000°C. Then the resultant quicklime (calcium oxide) is hydrated to slaked lime (calcium hydroxide) which sets by loss of water and then hardens over time by carbonation (the absorption of CO₂ from the atmosphere). According to BS EN459-part1: 2010 [2], hydrated limes are classified by calcium content. CL90 stands for calcium lime with at least 90% of calcium hydroxide.
Portland cement, which sets and hardens by hydration reactions, is made by heating a mixture of limestone and clay to 1500°C then grinding the resultant clinker to a fine powder with the addition of 3-4% of gypsum. Portland cement consists mainly of lime (CaO), silica (SiO₂), alumina (Al₂O₃) and iron oxide (Fe₂O₃). The four compounds which are usually regarded as the major constituents of cement are: tricalcium silicate (C₃S), dicalcium silicate (C₂S), tricalcium aluminate (C₃A) and tetracalcium aluminoferrite (C₄AF).

Between these two extremes of hydrated lime and Portland cement, there is a range of natural hydraulic limes (NHLs) which are produced by firing limestone, which contains naturally occurring clay impurities, at 900°C. Hydraulicity is defined as the ability of the material to set under water. The degree of hydraulicity of the resulting lime depends on the clay content of the parent limestone. The higher the percentage of clay impurities in the limestone is, the more the hydraulic the lime. Artificial hydraulic limes are made by mixing a pozzolanic additive to air lime in order to induce a hydraulic set. Cement and hydraulic limes set and harden by hydration reactions and carbonation makes a lesser contribution to strength gain [1, 3].

European Standard EN 459-part1: 2010 [2] characterizes hydraulic limes by the strength classes of 2, 3.5, and 5. The designator number after NHL is the minimum expected compressive strength in MPa of a 1:3 lime: sand mix achieved after 28 days under laboratory conditions. NHL2 is therefore the weakest and NHL5 the strongest.

These traditional building materials are porous and permeable and the movement of water through porous building materials has many important consequences in building construction, building maintenance and building use. In any cubic metre of brick, stone and concrete about one fifth is open porosity [4]. Water transport processes are not just important during the life of a building but are also critical during the construction stage. Water movement through porous materials, such as clay bricks, has a large influence on their performance, and is the main cause of many practical problems such as rising damp, frost damage, efflorescence, and sulphate attack in mortar. The total porosity of clay bricks has a major effect on compressive strength, and permeability. Thus, in the specification of clay bricks, water absorption is always taken as a guide for the prediction of durability. On the other hand the capillary suction properties of clay bricks also have an effect on both construction practice and weathering performance. The mix water abstraction from a freshly-mixed wet mortar by the substrate depends on both the capillary suction of the substrate and the water retentivity of the fresh mix [5]. This has significant influence on both workability and setting time in the freshly mixed...
mortar [6], and the resultant properties of the set and hardened mortar such as adhesion, strength (flexural and compressive) and sorptivity. Dewatering of freshly-mixed mortars, which is the major concern of this research, has been little studied and there is little published work on the properties of dewatered wet mixes.

This research project is an experimental study which has sound basis in theoretical background. The study is based on experimental work, which is the major part of the work, and literature review. There is a large amount of literature on mortars but not a lot on dewatered mortars. The number of journal articles is very large, but it is extremely difficult to find evidence of a fundamental approach.

A considerable time has been spent on the focus of the work. This work establishes a new method to examine the effect of dewatering on strength, sorptivity and setting time of cement and hydraulic lime mortars. This study is highly complementary to the current international effort in measuring and quantifying materials properties and is unique in the examination of the effects of dewatering on the properties of mortars in both the soft and hardening states. An assessment of the current standards related to water transport in porous building materials is needed, and therefore this study has the potential to input into new standards.

1.2 AIM OF STUDY

The overall aim of the work is to investigate the mechanism and effect of the dewatering of fresh mortar in contact with masonry materials. This leads to four subsidiary aims:

1. To gain a better understanding of water movement between mortar and brick in masonry structures and to investigate the properties of the dewatered state, in which mortar is neither freshly mixed nor set and hardened. This state is a temporary state which is sometimes short.

2. To establish, measure and quantify the effects of dewatering by absorbent substrates on the properties of mortars and renders in both the freshly mixed and the set and hardened states.

3. To examine the effect of pozzolanic addition on the water retaining ability of mortars and from this to examine the possibility of controlling the water retaining ability of the freshly-mixed mortar by mix composition and thus balance this with the suction properties of the substrate.
4. To examine and validate the Sharp Front equation \( S = (2Kf\Delta \Psi)^{1/2} \) by careful experimental measurements of the hydraulic parameters (including measurement of capillary pressure on selected bricks) and enable the work of this thesis to be applied to other dewatering systems such as sewage sludges and oil field slurries.

1.3 OBJECTIVES

In order to achieve these aims the following objectives have been undertaken:

**Aim 1 objectives**

- To investigate experimentally and develop the methodology of the American Petroleum Institute (API) pressure cell technique for measuring the water retaining ability (desorptivity) of freshly-mixed wet mortar.
- To investigate the statistical precision of API pressure cell method by calculating repeatability standard deviation.
- To examine the water retaining ability of freshly-mixed hydraulic lime and cement mortars and to demonstrate the role of factors such as hydraulicity, additive materials, binder particle size and the mix water chemistry on the water retentivity of these mortars.
- To investigate experimentally the main properties of water transport in fired clay brick: capillary suction and water absorption porosities.

**Aim 2 objectives**

- To design a brick mould to produce mortar bars of fixed dimensions that had been dewatered prior to setting.
- To investigate experimentally the effect of dewatering, by the capillary suction of common clay facing bricks, on the setting time, sorptivity and strength of mortars prepared from natural hydraulic lime NHL5 and Portland cement CEMI.

**Aim 3 objectives**

- To investigate the effect of the non-pozzolanic addition of limestone powder on the desorptivity of Portland cement mortar and the effect of the addition of a range of volume fractions of the pozzolana ground granulated blast furnace slag (GGBS) on the desorptivity of NHL5 mortars.

**Aim 4 objectives**

- To design and develop a sorptivity measurement method by visual inspection of the wet front capillary rise.
• To design and develop a sorptivity measurement under vacuum to eliminate the resistance of air pressure on the capillary suction of clay bricks.
• To compare the calculated value of the capillary pressure from measurement of its defining parameters with the measured value.

General objectives
• To review relevant research literature and theoretical background on the topic.
• To undertake a detailed review and evaluation of the available test methods for water desorption.
• To critique the standard tests.
• To write up and present work and to produce scientific publications in international conferences and journals.

Relationship between objectives
The objectives for aims 1, 2, 3 and 4 form four independent work-streams, which link to the overall aim and all contribute to the overall conclusions.

1.4 THESIS STRUCTURE
This thesis consists of nine chapters in which five of them report experimental work. After the current introductory chapter, a theoretical background and literature review is presented in chapter 2. The first part of this chapter includes a theoretical background of water transport in porous materials. The related theories and concepts defining the saturated and unsaturated flow in porous materials are described. The second part of chapter 2 mainly focuses on the practical background. The dewatered state of freshly mixed mortars is discussed although there is little published research on this subject. A comprehensive review and evaluation of the available test methods for water retention is presented and, in the final section, the applications of statistical methods for the accuracy of measurement methods are discussed.

Chapter 3 summarises the materials and the main experimental techniques used in the experimental work. Clay bricks, the binder materials (Portland cement and natural hydraulic limes) and the pozzolanic additive ground granulated blast furnace slag (GGBS) are described in detail. Experimental techniques, both standard and developed, used to measure properties of the solid material and of mortars in the freshly-mixed wet state are described. A detailed explanation of the preparation of brick moulds used to dewater mortars is given.
The main experimental work is presented in chapters 4 to 8. A comprehensive investigation of the reliability, applicability and accuracy of the methodology of the American Petroleum Institute (API) pressure cell technique for testing the water retaining ability of fresh mortar is presented in chapter 4. The evaluation of the API pressure cell technique was carried out in two parts: first by changing the controlled variables of the experimental set-up. These include the time interval for the collection of experimental data, the mass of mortar in the pressure cell and the pressure of the system. Secondly the consequences of the results obtained were discussed. These include gas breakthrough time and the water content of the resultant filter cake.

In chapter 5 the water retaining ability of freshly mixed hydraulic lime and cement mortars was examined and the role of hydraulicity, additive materials (pozzolanic and non-pozzolanic), binder particle size and the chemistry of mix water on the water retentivity of these mortars was investigated. The effect of the addition of non-pozzolan i.e limestone powder and a range of volume fractions of the pozzolan ground granulated blast furnace slag (GGBS) on the desorptivity of cement and hydraulic lime mortars was examined. The particle size distributions of six binder materials as well as mixes with GGBS were measured and the effects of particle size on desorptivity discussed. The effect of sulphate solution of the same concentration as CEM I extract used as the mix water on the desorptivity of hydraulic lime mortar (with and without GGBS) was investigated.

Chapter 6 is an experimental comparison of the main properties of water transport in three types of fired clay brick: capillary suction and water absorption porosities. Sorptivity for three faces (bed, stretcher, and header) of each brick type was determined and the uniformity of the bricks was discussed. The initial rate of absorption (1 minute test) was measured and compared with the sorptivity. The water absorption for different soaking times from 1 minute to many hours, satiated porosity and vacuum saturation porosity test methods were discussed and compared for three types of clay brick. The time for water to travel to the centre of the brick and the proportion of saturation achieved using water absorption test results were calculated and presented.

After Chapters 5 and 6 have considered the water transport properties of mortars and substrates (clay bricks) individually, Chapter 7 considers the movement of water between the two in combination as in construction. This chapter reports an experimental study to investigate the effect of dewatering, by the capillary suction of common clay facing bricks, on the setting time, sorptivity and strength of freshly-mixed mortars prepared from natural hydraulic lime NHL5 and Portland cement CEM I. The time to
dewater a fixed depth of mortar and the depth of brick needed were calculated using data from previous research. An absorbent brick mould was designed to produce mortar bars of 40x40x160 mm which had been dewatered prior to setting.

Chapter 8 presents an experimental validation of a fundamental Sharp Front equation. A comprehensive measurement of sorptivity, permeability, porosity and capillary pressure of two types of clay brick were obtained. Developed techniques and conventional methods were used to measure sorptivity and porosity. Saturated permeability was measured by the simple constant head method. Capillary pressure was obtained by measuring the equilibrium pressure of the air compressed ahead of the advancing wetting front. Data of sorptivity, saturated porosity and permeability lead to the calculation of capillary pressure which is hard to measure experimentally. Values of measured capillary pressure were obtained and compared with the calculated values and the closeness to calculated values taken as validation of the equation.

Finally, chapter 9 presents the main conclusions of the study and recommendations for future work.
CHAPTER 2  THEORETICAL BACKGROUND AND LITERATURE REVIEW

2.1 INTRODUCTION
The first part of this chapter provides the theoretical background of water transport in porous materials discussing both saturated and unsaturated flow. The chapter begins with a definition of the main properties that are related to water movement in porous building materials. The relevant theories and concepts defining the flow in porous materials are described. Darcy’s law and Sharp Front theory are used to derive equations describing sorptivity, desorptivity and transfer sorptivity. The relationship between these parameters is described. Moreover an equation describing the time taken to dewater a mortar joint is derived.

The second part of the chapter is a review of the practical background. This part has three sections. It begins with a definition of freshly mixed mortar and its main constituents. The important practical properties of fresh mortars are discussed and a review of studies on these properties is included. The dewatered state of freshly mixed mortars is discussed although there is little published research on this subject. The effect of dewatering on compressive strength and setting time of freshly mixed cement and lime mortars is reviewed. Pozzolanic and non-pozzolanic materials and their effects on the water retentivity of fresh mortar are discussed. The second section provides a comprehensive review and evaluation of the available test methods for the measurement of the water retentivity of freshly-mixed mortars. In third section the applications of statistical methods for the accuracy of measurement methods are discussed.
Chapter 2: Theoretical Background and Literature Review

2.2 Theoretical Background of Water Transport in Porous Materials

Most inorganic building materials such as brick, stone and concrete are porous and permeable to water vapour and liquid water. It is well known that many of the durability problems in building construction are related to water transport in porous building materials. This section gives the theoretical background and definitions of the main parameters of hydraulic flow in porous building materials.

2.2.1 Porosity

The term porosity relates to the total volume of voids in a material. It is defined as the ratio of the volume of the pore space /bulk volume of the material [7]. Porosity so defined has the symbol \( f \) and it is given as a \( \% \) by volume. Traditional building materials have porosities ranging between 0.4 and 0 for fired clay bricks and marble and granite respectively [7]. Materials which are made by generating gas bubbles within the paste or during firing usually have high porosity. An example of this is Aerated Autoclaved Concrete block (AAC). These materials have microstructures different from those of sintered or cementitious materials. A process such as sintering act to close or isolate some of the pore space and as a result open porosity reduces because parts of the pore space become disconnected.

Any closed porosity does not contribute to the transport of gases and liquids and it is not counted in the porosity measured in this thesis. It is important to distinguish between open and closed, or connected and disconnected porosity. Closed porosity is very difficult to measure and is often counted as part of the solid mass. The existing methods for measuring porosity involve penetration of gas or liquid into the pore system (liquid saturation or gas saturation). Therefore these methods determine open porosity and do not include any closed porosity. There are various test procedures used to measure open porosity which are presented and compared in [8]. Vacuum saturation porosity \( f \) represents the absorption of water by immersion under vacuum [9] and satiation porosity \( f' \), which is also termed “effective porosity”, represents the absorption of water by capillary rise.
2.2.2 Permeability

Darcy’s law of saturated flow

The definition of permeability in porous materials is based on Darcy’s law which describes permeability, \( k \) as the movement of liquid through a saturated porous material under the action of a pressure gradient. Saturated flow is pressure-driven rather than absorption-driven due to the constant water content of the saturated medium. Darcy’s law for one-dimensional flow in a saturated porous material is expressed by the following equation [7]:

\[
Q = -\frac{k A_s \Delta P}{L}.
\]  

(2.1)

This states that the volumetric flow rate \( Q \) of liquid through a specimen of porous material is directly proportional to the hydrostatic pressure difference \( \Delta P \) across the specimen, inversely proportional to the length \( L \) and directly proportional to the cross sectional area \( A_s \). The constant of proportionality is the Darcy permeability of the saturated material \( k \).

As construction materials are the major focus of this study, it should be noted that they are rarely completely saturated either during construction or throughout their lifetime. Hall, 1977 [10] describes saturated flow as the limiting case of the more general unsaturated flow. Darcy’s law was extended to apply to unsaturated capillary flow and was first developed in soil physics [11], but in this case the Darcy saturated permeability is replaced by a general flow property called the hydraulic conductivity which is strongly dependent on the water content \( \theta \). Hydraulic conductivity is usually denoted as \( K \).

Saturated permeability can be determined directly according to the simple Darcian definition of flow. The preferred experimental arrangement to measure saturated permeability is by the steady flow of water through a cylindrical specimen of uniform cross-sectional area and sealed circumferential surface under the action of a known pressure head. Two experimental arrangements developed in the Construction Science Laboratory at Manchester are widely used to measure the permeability of granular materials and rigid solids. One is the constant head permeameter which measures permeability at a constant pressure head and the other is the falling head permeameter which measures the flow rate at a reducing pressure head [12]. Figure 2.1 shows a schematic diagram of the principles of both constant and falling head permeability measurement.
In the constant head method flow rate is determined by weighing the outflow at intervals while the water level is kept constant at $h_0$. The permeability is obtained from Darcy’s law:

$$Q = kA_s h_0 / L$$  \hspace{1cm} (2.2)

where $A_s$ is the cross sectional area of the sample. It is preferable that measurements are made for a range of values of $h_0$. The flow rate should vary linearly with $h_0$ if the flow is Darcian.

The falling head method is unlike the constant head method. During measurement the level of water in the tube falls a distance $h$ from the initial position $h_0$ to some height $h_t$ over a time interval $t$ as shown in figure 2.1. The flow rate at any height $h_t$ is given by the following equation [12]

$$Q = A_c \frac{\Delta h}{\Delta t}$$  \hspace{1cm} (2.3)

where $A_c$ is the cross-sectional area of the water column. Applying Darcy’s law to this flow process gives:

$$\ln \frac{h_0}{h_t} = \frac{kA_s}{LA_c} t .$$  \hspace{1cm} (2.4)

A graph of $\ln \frac{h_0}{h_t}$ against $t$ will produce a straight line if the flow is Darcian and from the gradient of the graph the permeability $k$ may be calculated from:

$$k = m L \frac{A_c}{A_s} .$$  \hspace{1cm} (2.5)

Both constant and falling head permeameters can be used for most porous building materials including mortars. For very low permeability materials (such as some types of engineering brick, stone or concrete) a relatively high driving pressure is required to achieve measurable flow. A permeameter which is suitable for such high pressure
permeability is the Hassler cell. This was designed and developed in Manchester to contain cylindrical cores of 23-25 mm in diameter and 25-75 mm in length. The saturated core is enclosed in a sleeve of nitrile rubber to which a containing pressure is applied to seal the specimen and ensure unidirectional flow. A chromatography pump provides a pulse-free constant flow rate of liquid through the sample and this flow pressure is recorded. A schematic diagram of the Hassler cell is shown in figure 2.2 and full description of the experimental technique and associated equipment is given in Green et al. [13].

Fig 2.2: Hassler cell permeameter [14].

The Hassler cell is widely used in petroleum technology [15] and has been used to obtain permeability values for construction materials [16-19].

Darcy’s law applies also to the flow of gases through porous materials but in this case there are some differences in the equations for gas permeability because of gas compressibility. For gas permeability the mass of gas is measured because it is constant whereas because gas is compressible, its volume is not. For accurate determination of gas flow in rocks, a correction factor of gas slippage called the Klinkenberg correction
Chapter 2: theoretical background and literature review

is usually used because gas permeability is higher than liquid permeability [15, 20]. For cement-based materials previous work [21, 22] has shown that there is a significant material change due to the hydration of unreacted cement caused by contact with water. Therefore permeability measured with gas (air or nitrogen) is often preferable although the value is higher than that measured using water. Alternatively, the permeability to organic liquids may be used.

2.2.3 Capillary pressure

Porosity and saturated permeability do not tell the whole story of water transport in porous materials. Another important property is the capillary pressure $\Psi$ or capillary potential, which for this study is equivalent to the pressure of trapped gas in the wetted region of an unsaturated material which is described later. $\Psi$ is difficult to measure experimentally. The capillary suction property of any porous material is characterized by its capillary pressure function. Capillary pressure is defined as the energy required to transfer unit mass of liquid from a partially saturated porous material to a reservoir of the same liquid at the same temperature and elevation [20]. Water movement in porous building materials is almost completely driven by the action of capillary forces. Ioannou et al. [23] described a new experimental method of measuring directly the capillary pressure $\Psi$ of a wetting front. They showed that the capillary pressure is a material property and it defines the effective strength of the capillary forces that drive the absorption in a porous material. The method measures the equilibrium pressure of the air displaced and compressed ahead of the advancing wetting front. The pressure of air trapped in the wetted region is taken as the capillary pressure of absorption, using the assumptions of the ideal gas law. These assumptions and the experimental difficulties limit the method, but it is the only simple, direct method of measuring $\Psi$.

2.2.4 Development of the general unsaturated flow equation

This analysis based on Hall and Hoff [7], but is adapted to be clarified for the derivation of sorptivity, desorptivity, transfer sorptivity and time to dewater in sections 2.2.6 and 2.2.7.

Darcy’s one-dimensional saturated flow equation was developed in soil physics and has been applied to the movement of water in building materials in which the water content is less than saturation. A schematic is shown in figure 2.3.
Fig 2.3: Schematic representation of unsaturated flow.

The following equation was developed from the saturated case in which the flow is pressure-driven rather than absorption driven.

\[
Q = -\frac{K A_s (h_2 - h_1)}{z_2 - z_1}
\]  

(2.6)

where
- \(Q\) Quantity of water flowing through the sample per unit time (mm\(^3\)/min)
- \(K\) Hydraulic conductivity (mm/min)
- \(A_s\) Cross section area of sample (mm\(^2\))
- \(h_2 - h_1\) Hydraulic head (mm)
- \(z_2 - z_1\) Thickness of sample (mm)

The negative sign in equation (2.6) is used so that a positive value of \(Q\) will indicate flow (downward) from \(h_2\) to \(h_1\) [24]. The positive direction (upward) is measured from \(z_1\) to \(z_2\). The hydraulic gradient is \(\frac{h}{L}\). The ratio \(\frac{Q}{A}\), the flux per unit cross sectional area, is also known as the Darcy velocity, \(u\), which is given by

\[u = \frac{Q}{A}.
\]  

(2.7)

Substituting equation (2.7) into (2.6) gives

\[u = -\frac{K h}{L}.
\]  

(2.8)

In differential form and in three dimensions equation (2.8) may be expressed as

\[u_x = -K \frac{dh}{dx},
\]  

(2.9)

\[u_y = -K \frac{dh}{dy}.
\]  

(2.10)
and
\[ u_z = -K \frac{\partial h}{\partial z} \]  
(2.11)
in the x, y and z directions respectively.

Laplace’s continuity equation which states mathematically that mass can neither be created nor destroyed [24] can be written as:
\[ \frac{\partial \theta}{\partial t} = -\left( \frac{\partial u_x}{\partial x} + \frac{\partial u_y}{\partial y} + \frac{\partial u_z}{\partial z} \right) \]  
(2.12)
where \( \theta \) is the volume fraction water content and \( u_x, u_y \) and \( u_z \) are the Darcy velocities in the x, y, and z directions.

Equation (2.12) states that water is conserved in the system. This means that in any volume element at any time the amount of water present is equal to the original amount plus the amount entering the element minus the amount leaving.

Substituting equation (2.9), (2.10) and (2.11) into equation (2.12) and remembering that \( h \) is hydraulic head measured upward from an arbitrary reference level gives
\[ \frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x} \left( K \frac{\partial h}{\partial x} \right) + \frac{\partial}{\partial y} \left( K \frac{\partial h}{\partial y} \right) + \frac{\partial}{\partial z} \left( K \frac{\partial h}{\partial z} \right). \]  
(2.13)
Writing the total head \( h \) as
\[ h = h_t + z \]  
(2.14)
where \( h_t \) is the tension head and \( z \) is the gravitational head [24] gives the general form of the unsaturated flow equation
\[ \frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x} \left( K \frac{\partial h_t}{\partial x} \right) + \frac{\partial}{\partial y} \left( K \frac{\partial h_t}{\partial y} \right) + \frac{\partial}{\partial z} \left( K \frac{\partial h_t}{\partial z} \right) + \frac{\partial k}{\partial z}. \]  
(2.15)

\( h_t \) is the hydraulic tension head which can be sustained by capillary rise from pores of diameter equal to the mean diameter of the pores at the air-water interface [25]. Figure 2.4 shows a schematic of the idealized pore interface.

![Figure 2.4: The tension head, h_t, of pore water [25].](image-url)
Equation 2.15 can be separated into flows in the x, y and z direction:

\[ K \frac{\partial h_t}{\partial x} = K \frac{\partial h_t}{\partial \theta} \frac{\partial \theta}{\partial x}, \]  
(2.16)

\[ K \frac{\partial h_t}{\partial y} = K \frac{\partial h_t}{\partial \theta} \frac{\partial \theta}{\partial y}, \]  
(2.17)

and

\[ K \frac{\partial h_t}{\partial z} = K \frac{\partial h_t}{\partial \theta} \frac{\partial \theta}{\partial z}, \]  
(2.18)

Considering the x direction only:

\[ u_x = -K \frac{\partial h_t}{\partial x} \]  
(2.19)

Allows the diffusivity, \( D \), to be defined by

\[ D = K \frac{\partial h_t}{\partial \theta}. \]  
(2.20)

Therefore, for horizontal flow in the x and y directions:

\[ u_x = -D \frac{\partial \theta}{\partial x}, \]  
(2.21)

\[ u_y = -D \frac{\partial \theta}{\partial y}, \]  
(2.22)

and for vertical flow in z direction

\[ u_z = -D \frac{\partial \theta}{\partial z} - c \]  
(2.23)

where \( c \) is a retardation effect due to gravity.

The general form of the unsaturated flow equation considering only horizontal flow in the x direction eq. (2.15) is

\[ \frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x} \left( K \frac{\partial h_t}{\partial \theta} \frac{\partial \theta}{\partial x} \right). \]  
(2.24)

Substituting for \( D \) from equation (2.20) in equation (2.24) gives

\[ \frac{\partial \theta}{\partial t} = \frac{\partial}{\partial x} \left( D \frac{\partial \theta}{\partial x} \right). \]  
(2.25)

\( D \) is a function of water content \( \theta \) such that

\[ D(\theta) = D_0 e^{B\theta} \]  
(2.26)

where \( D_0 \) and \( B \) are constants. \( D_0 \) is effectively a diffusion coefficient. This analysis shows that hydraulic diffusivity is comparable to thermal diffusivity in that they both involve flow.

### 2.2.5 Sharp Front theory

Sharp Front (SF) models given by Green and Ampt 1911, Philip 1969, Hall and Hoff 2002 [7, 26, 27] provide a powerful, simplified but approximate approach to the analysis of unsaturated capillary transport of liquids in porous media. Sharp Front theory of liquid absorption processes assumes that the wetted region behind the wet front is uniform and has constant water content and that the position of the advancing
wet front is marked by a step change in liquid content. Hall and Hoff, 2002 [7] stated that the capillary absorption profiles of porous materials are often very steep because the capillary diffusivity $D$ differs greatly with water content $\theta$. Figure 2.5 shows schematically the concept of Sharp Front theory as applied to the capillary water absorption by an initially dry porous solid.

Figure 2.5: Schematic representation of the SF theory approach shown in (a) and the actual capillary water absorption in (b) in a porous solid. The water content ($\theta$) versus distance profile is shown in each case.

$X'$ represents the end of the wet region or front. Figure 2.5 (a) shows the principle of the Sharp Front model. The profile of the water content versus distance is sharp in the idealized case therefore the SF approach is to represent the wetted region by a rectangular block. In reality the wetted region is diffuse, the point in contact with water being saturated, and the water content reducing towards the wet front as shown in figure 2.5 (b). The water content $\theta$ versus distance $X$ profile is shown in each case. Areas A and B are considered to be equal. This is called the Sharp Front approximation which reduces unsaturated flow problems.

Section 2.2.6 will show that SF theory can be applied to the absorption of water by a porous solid from a free water surface to derive the sorptivity; to the absorption from a
freshly mixed wet mortar to derive the transfer sorptivity; and can also be applied to the dewatering of freshly mixed mortar to derive the desorptivity.

2.2.6 Sharp Front theory applied to porous solid materials

In the following section it is assumed that there is no reaction between the porous solid and the liquid (water) flowing through it.

2.2.6.1 Derivation of sorptivity

SF theory and Darcy’s law are applied to the absorption of free water by porous solids to derive the sorptivity. Gummerson et al. [28] defined sorptivity as a measure of the ability of a porous material to absorb and transmit water or other wetting fluid by capillarity. It is well established [28-31] that the sorptivity $S$ is the most useful parameter to describe the water absorption properties of a porous solid. It is also extensively used in concrete technology although it is not yet included in either the British Standard or the RILEM water absorption test procedures [17]. The one-dimensional advance of water into a vertical bar of porous material is shown in figure 2.6.

![Figure 2.6: Schematic of SF absorption into a porous solid showing the parameters used in the derivation of sorptivity [7].](image)
In SF theory the capillary pressure $\Psi_1$ at the wet front is considered to be constant and the capillary pressure $\Psi_0$ at the inflow face is zero at $x = 0$. Applying Darcy’s law gives the following equation

$$\frac{dQ}{dt} = -KA \frac{\Delta \Psi}{x} \quad (2.27)$$

where $\Delta \Psi$ is the negative value of the capillary potential differential across the advancing length $x$ and $\frac{dQ}{dt}$ is the volume of water absorbed per unit time. The flow contained within the wetted region is given by $Q = fA_s x$ where $f$ is the volume fraction porosity, $A$ is the cross sectional area and $x$ is the length of the wetted region and therefore

$$dQ = fA_s dx. \quad (2.28)$$

Substitution of equation (2.27) into (2.28) gives

$$fA_s \frac{dx}{dt} = -KA_s \frac{\Delta \Psi}{x}. \quad (2.29)$$

Rearranging equation (2.29) gives

$$xdx = \left(-\frac{K\Delta \Psi}{f}\right) dt. \quad (2.30)$$

Integration of equation (2.30) gives

$$\frac{x^2}{2} = \left(-\frac{K\Delta \Psi}{f}\right) t + C \quad (2.31)$$

where $C$ is the constant of integration. When $t = 0, \ x = 0$ therefore $C = 0$.

Rearranging equation (2.31) gives

$$x = \left(-\frac{2K\Delta \Psi}{f}\right)^{1/2} t^{1/2}. \quad (2.32)$$

Equation 2.32 shows that the advance of the wet front is proportional to $t^{1/2}$. Substituting for $x$ from equation (2.32) into (2.27) gives

$$\frac{dQ}{dt} = -K A \Delta \Psi \frac{f^{1/2}}{(-2K\Delta \Psi)^{1/2}} t^{-1/2}. \quad (2.33)$$

Simplifying equation (2.33) gives

$$\frac{dQ}{dt} = K^{1/2} A \Delta \Psi^{1/2} \frac{f^{1/2}}{\sqrt{2}} t^{-1/2}. \quad (2.34)$$

Multiplying the numerator and denominator of equation (2.34) by $\sqrt{2}$ and rearranging gives

$$\frac{dQ}{dt} = \frac{1}{2} (2KA^2 \Delta \Psi f)^{1/2} t^{-1/2}. \quad (2.35)$$

Integrating equation (2.35) gives

$$Q = (2KA^2 \Delta \Psi f)^{1/2} t^{1/2} + C' \quad (2.36)$$
where \( C' \) is the constant of integration. Again at \( t = 0, Q = 0 \) so \( C' = 0 \).

The sorptivity \( S \) is now defined by

\[
S = (2KΔΨf)^{1/2}.
\]

(2.37)

Substituting for \( S \) in equation (2.36) gives

\[
Q = SAT^{1/2}.
\]

(2.38)

Since \( \frac{Q}{A} \) is the volume of water absorbed per unit area of absorbing surface, it is denoted by \( i \) and thus defined

\[
i = St^{1/2}.
\]

(2.39)

It can be seen that the total volume of liquid absorbed per unit area by capillarity, \( i \), increases linearly with the square root of time.

Since \( i \) is easily measured at any value of \( t \), equation (2.39) is used to determine \( S \) as the gradient of the graph of \( i \) versus \( t^{1/2} \) in experimental measurement of the capillary absorption of water into the surface of an initially dry porous sample.

### 2.2.7 Sharp Front theory applied to the desorption of water from freshly-mixed wet mortars

#### 2.2.7.1 Derivation of desorptivity

Water retentivity is the ability of a freshly-mixed wet mortar to retain its mix-water when placed in contact with an absorbent substrate. In 2002, Hall and Hoff [7] derived the theoretical basis for and quantified the water retaining ability of freshly-mixed wet mortars by a well-defined parameter, the desorptivity \( R \). A high value of \( R \) corresponds to a wet mix which is very water releasing. A low value of \( R \) corresponds to a wet mix which is very water retaining. Sharp Front theory is used in the derivation of desorptivity. It is assumed that in the dewatering process the wet mix forms a filter cake, and that both the wet mix and the filter cake have uniform initial and final water contents. As dewatering progresses there is a sharp front between the wet mix and the dewatered filter cake, the depth of which increases as \( t^{1/2} \) [7]. Figure 2.7 shows a schematic representation of the sharp front desorption of water from a wet mix and shows the parameters used in the derivation of desorptivity.
Figure 2.7: Schematic of sharp front desorption of water from a wet mix [7].

An enclosed column of wet mix of depth $L$ is caused to dewater under the action of an applied gas pressure, $P$.

Assuming the initial volume fraction of solids in the fresh mix is $\phi_i$ (which is considered to be uniform), and the initial volume fraction of water is $\theta_i$ then the total initial volume fraction of water and solid in the fresh mix is

$$\phi_i + \theta_i = 1. \quad (2.40)$$

As dewatering proceeds, a filter cake is formed which has a uniform solids content $\phi_c$ and a uniform water content $\theta_c$. The cumulative volume of desorbed water per unit area of wet mix $\iota$, is given by

$$\iota = L(\theta_i - \phi_i \theta_c / \phi_c) = L \alpha. \quad (2.41)$$

Where $\alpha$ is a variable that represents the initial and final volume fractions of both water and solids in the mortar and is defined by $\alpha = \theta_i - \phi_i \theta_c / \phi_c$. After dewatering, the initial depth $L$ of wet mix produces a filter cake of depth $L_c$ which is given by

$$L_c = L \phi_i / \phi_c = L \beta. \quad (2.42)$$

From equations (2.41) and (2.42), $L_c + \iota = L$, $\iota = L_c \alpha / \beta$ and $\alpha + \beta = 1$.

As dewatering proceeds, the cake thickness increases as the water is desorbed at a rate which is controlled by the permeability $K_c$ of the cake [7].

Applying Darcy’s law to the dewatering process gives

$$\frac{di}{dt} = -K_c \frac{\Delta P}{L_c} = -K_c \frac{\alpha \Delta P}{\beta \iota} \quad (2.43)$$
where $P$ is the dewatering pressure. Integrating equation (2.43) with respect to time $t$ gives

$$i = \left(\frac{2K_c \Delta \rho \alpha}{\beta}\right)^{1/2} t^{1/2}. \quad (2.44)$$

From equation 2.44 the desorptivity is defined by

$$R = (2K_c \Delta \psi \alpha / \beta)^{1/2}. \quad (2.45)$$

From equations (2.44) and (2.45), the equation defining desorptivity is given by

$$i = Rt^{1/2}. \quad (2.46)$$

Experimentally the desorptivity $R$ of a fresh mix is obtained from the gradient of a plot of $i$ versus $t^{1/2}$. In this case $i$ is the volume of water desorbed per unit cross-sectional area of wet mix.

**2.2.7.2 Derivation of transfer sorptivity**

Transfer sorptivity ($A$) is a measure of the absorption of water by a porous solid (such as brick) from a freshly mixed wet slurry such as mortar. In principle it is the same as sorptivity except that the absorption of water is not from a free water surface but from freshly mixed wet mortar. In the derivation of transfer sorptivity, SF theory may be applied to both the absorption of water by a porous solid and also to the desorption of water from a wet mix. In this case both the sorptivity of the porous solid and the desorptivity of the wet mix are involved. Figure 2.8 shows the parameters for dewatering of a wet mix by an initially dry porous material. The wet mix in figure 2.8 has a volume fraction of solid $\theta_i$ and a volume fraction of water $\theta_i$.

![Figure 2.8: Capillary absorption of water by a porous solid in contact with a wet mix.](image)
After dewatering, the filter cake has its own volume fraction of solids $\phi_c$ and a volume fraction of water $\theta_c$. It must be noted that the summation of $\phi_i + \theta_i = 1$ applies to the wet mix. The absorption of water from the freshly mixed wet mortar by the dry absorbent substrate is by capillary action. This process results in the formation of a wetted zone in the absorbent substrate as shown in figure 2.9.

SF theory assumes that the water content is uniform behind the wet front. As the water moves from the wet mix to the absorbent substrate the solid particles in the wet mix are moved to the wet mix/substrate interface. Finally an incompressible filter cake is formed as these solid particles settle together at the interface. As desorption proceeds, the volume fraction of solids in the filter cake $\phi_c$ increases and the volume fraction of water $\theta_c$ decreases.

The initial length of the wet mix is $L$ and as desorption proceeds this length is converted to filter cake. When all the mix has been converted to filter cake desorption stops and the thickness of the filter cake $L_c$ can be determined from:

$$L_c = L \frac{\phi_i}{\phi_c} = L\beta.$$  \hspace{1cm} (2.47)

As water is withdrawn from the wet mix the length of the wetted zone in the absorbent substrate $L_w$ increases. At the same time the depth of the filter cake $L_c$ increases and is proportional to $L_w$. The ratio $L_c / L_w$ therefore always remains constant, (given by $C$), as long as desorption proceeds. The volume of water contained in the wetted zone of the

---

**Figure 2.9:** Schematic representation of SF absorption of water from wet mix by a porous solid [5].
absorbent substrate is the same as the volume of water desorbed from the wet mix. It follows from this that \( \frac{i}{L_c} = \frac{\infty}{\beta} \) and \( \alpha + \beta = 1 \).

The length of the wetted zone can be expressed by \( L_w = \frac{i}{f} \), where \( f \) is the porosity of the substrate. Therefore, \( L_c = L\beta \) and the constant \( C \) can be written as

\[
C = \frac{L_c}{L_w} = \frac{f\beta}{\alpha} \quad (2.48)
\]

The volume of water contained in the wetted zone of the absorbent substrate, \( i \), is

\[
i = L \left( \theta_i - \frac{\theta_i}{\theta_c} \right) = L\alpha \quad (2.49)
\]

Applying Darcy’s law to the whole system shown in figure 2.10 gives

\[
\frac{di}{dt} = -K_{\text{eff}} \frac{\psi_f}{L_c + L_w} = -K_{\text{eff}} \frac{\psi_f}{i(C+1)} \quad (2.50)
\]

where \( K_{\text{eff}} \) is the permeability of the whole system.

Since \( i = 0 \) when \( t = 0 \), integration of equation (2.50) gives

\[
i = \left( \frac{2K_{\text{eff}}\psi_f}{C+1} \right)^{1/2} t^{1/2} \quad (2.51)
\]

From equation (2.51) the transfer sorptivity \( A \) is defined by

\[
A = \left( \frac{2K_{\text{eff}}\psi_f}{C+1} \right)^{1/2} \quad (2.52)
\]

and the final equation defining transfer sorptivity is given by

\[
i = At^{1/2} \quad (2.53)
\]

Experimentally, transfer sorptivity is obtained from the gradient of a plot of the cumulative absorbed volume of water per unit area of the substrate in contact with wet mix \( i \) versus \( t^{1/2} \).

### 2.2.7.3 Relationship between \( S, R \) and \( A \)

The relationship between sorptivity, \( S \), desorptivity, \( R \), and transfer sorptivity, \( A \), is given by [7]

\[
\frac{1}{A^2} = \frac{1}{R^2} + \frac{1}{S^2} \quad (2.54)
\]

Hall and Hoff (2002) gave the full derivation of this equation and it has been experimentally validated by Collier et al. [5] for the case of clay brick absorbing water from freshly mixed hydraulic lime and cement mortars. Ince et al. [32] extended this work and applied SF theory to derive an equation describing the time taken to dewater a wet mix by an absorbent substrate. This work also compared the calculated time to dewater with the experimentally measured time to dewater carried out on a range of
lime and cement mortars applied to a typical clay brick substrate. The derivation of the time to dewater is given in the next section.

### 2.2.7.4 Derivation of time to dewater

Ince *et al.* [32] developed a theoretical model to define the time to dewater a freshly mixed mortar bed \( t_{dw} \). Experimental measurements were carried out to examine the factors affecting the time to dewater and the analytical model was validated. SF theory was applied to derive an equation describing the time taken to dewater a wet mix, \( t_{dw} \), by an absorbent substrate. The theory assumes the brick-mortar-brick joints as shown in figure 2.10. Figure 2.10 (a) represents two bricks placed in contact with freshly mixed mortar before the start of dewatering.

![Figure 2.10: Schematic representation of the dewatering of a fresh mortar joint in brickwork: (a) immediately prior to dewatering; (b) on completion of dewatering at \( t = t_{dw} \) [32].](image)

As the bricks are initially dry the volume fraction water content of each, \( \theta_i \), is zero at time \( t = 0 \). The thickness of mortar is \( L \). It has an initial volume fraction water content \( \theta_i \) and volume fraction solids content \( \phi_i \) so that

\[
\theta_i + \phi_i = 1 \tag{2.55}
\]

The solids are assumed to be uniformly distributed throughout the wet mix. Bricks start to absorb water from either side of the wet mix by capillary suction and as a result a wet zone will advance in each brick. SF theory assumes that these wetted zones have uniform water content \( \theta_w \) as shown in figure 2.10 (b). As water continues to be withdrawn by the bricks, the solids in the wet mix are moved to the brick-mortar interfaces where they consolidate to form a filter cake. Desorption of water will stop when the entire mortar joint is dewatered and converted to filter cake and the wet front has travelled a distant \( L_w \) into each brick as shown in figure 2.10 (b). The resulting filter
cake will have a volume fraction water content, $\theta_c$, which is lower than that of the original wet mix and, in contrast, a higher volume fraction solid content, $\phi_c$. The thickness of the resulting filter cake will be reduced to $L_c = L \frac{\phi_i}{\phi_c}$ due to consolidation.

At the end of desorption the total volume of water per unit area desorbed from the wet mix is given by

$$ i = (L\theta_i - L_c\theta_c). \quad (2.56) $$

Also,

$$ L_w = \frac{i}{f} \quad (2.57) $$

where $f$ is the satiation volume fraction porosity of the brick [33].

Substitute $L_c = L \frac{\phi_i}{\phi_c}$ into equation (2.56) gives $i$ as:

$$ i = L \left( \theta_i - \phi_i \frac{\theta_c}{\phi_c} \right) = L\alpha \quad (2.58) $$

where $\alpha = \theta_i - \phi_i \frac{\theta_c}{\phi_c}$.

The earlier equation 2.53 described the suction of water from a wet mix by an initially dry substrate by

$$ i = At^{1/2} $$

where $A$ is the transfer sorptivity. When $L\alpha = At^{1/2}$, all the wet mix will be converted to cake and no further water can be abstracted by the substrate. The time at which desorption ceases, $t_{dw}$, is given by:

$$ t_{dw} = \left( \frac{L\alpha}{A} \right)^2. \quad (2.59) $$

Equation (2.59) shows that $t_{dw}$ increases proportionally with the thickness of mortar bed $L$, and with the parameter $\alpha$, a variable which embodies the initial and final volume fraction water and solid contents of the mortar. The time to dewater also depends on the transfer sorptivity $A$, and is in inverse proportion to it.

### 2.3 LITERATURE REVIEW

This part of the chapter firstly discusses the practical background of the main topic of this study which is the water movement between mortar and brick and the properties of the material in the dewatered state; and second gives a comprehensive review of the water desorption test methods. The last section discusses briefly statistical methods for the precision of API pressure cell method.
2.3.1 Practical background

2.3.1.1 Freshly mixed mortar

Mortar is defined by the Technical Dictionary of European Mortars in correspondence with EN and ISO standards, as “a mix of one or more inorganic or organic binders, aggregates, water, fillers, additives and/or admixtures” [34]. Standard BS EN 998-2 [35] defines binder as a material used to hold solid particles together in a coherent mass. In the work presented in this thesis the binder cement or lime is used with sand as aggregate. Aggregate is defined as the granular material that does not contribute to the hardening reaction of the mortar. A mortar is called a “masonry mortar” if it is used for bedding, jointing or pointing of masonry units [35]. Other types of mortar are rendering and plastering mortars.

An additive is a finely divided inorganic material that may be added to mortar in fairly large amount in order to improve or achieve special properties. An admixture is an organic or inorganic material added in small quantity to modify the properties of the mortar in the fresh and/or hardened state.

The primary function of mortar in masonry is to take up tolerances between building units such as brick or blocks. Mortars also have to fulfil some other requirements such as providing enough strength to resist frost or other forms of environmental attack. Mortars should resist the penetration of water and they also contribute to the aesthetic appearance of the wall [1]. In the fresh state mortars should be workable, yet cohesive. Mortar strength does not have a large effect on masonry strength since the mortar is applied in very thin layers (typically 10 mm).

There is a large amount of research reported on mortars in the hardened state but fresh mixes have been studied less. The important practical properties of fresh mortars that are of most concern to the work of this thesis are setting time and water retention ability. These properties are discussed in the following sections.

2.3.1.2 Setting time

Setting time is an important property of fresh mortar. For practical applications it is important to know when the material starts setting and when it finishes setting. Initial setting represents the end of workability and final setting is the beginning of hardening which is responsible for stiffness and strength development. Cement setting time reduces with a rise in temperature and at low temperatures setting is delayed [36, 37].
The most-used test by present cement manufacturers to measure the setting time of cement pastes and mortars is the Vicat needle test described in BS EN 196-3 (Methods of testing cement: determination of setting times and soundness) [38]. The physical background of the test is based on the resistance of a paste or mortar to the dynamic penetration of a rod (with a standard weight and shape) which will impose local shear stress. There is a large body of literature on cement setting time, but few publications on lime mortar setting time. Sleiman et al. [39] proposed a method which allows for the monitoring of the evolution of mortar setting, starting immediately from the mixing time, unlike the Vicat measurement which shows no changes until initial set. Reinhardt et al. [40] developed a testing device which utilizes the velocity of ultrasound waves to achieve continuous monitoring of the setting and hardening of cementitious materials with age. The results showed that the beginning of setting can be determined mathematically from velocity versus age of mortar curve. Trtnik et al. [41] studied the validity of the ultrasonic wave transmission method to estimate the initial setting time of cement paste. It was found that the initial setting time of a cement paste can be estimated precisely by the time at which the first inflection point appears on the ultrasonic pulse velocity curve. Karatasios et al. [42] studied the setting process of lime-based mortar with barium hydroxide as an additive. It was found that the setting and hardening of lime-barium mortars can be estimated from the carbonation of both calcium hydroxide and barium hydroxide. Hughes et al. [43] measured the initial and final setting times of 6 different hydraulic lime pastes each prepared with a different water content so as to produce a standard consistency. The results are shown in table 2.1.

<table>
<thead>
<tr>
<th>Limes</th>
<th>W/L ratio</th>
<th>Initial Set</th>
<th>Final Set</th>
</tr>
</thead>
<tbody>
<tr>
<td>St Astier NHL2</td>
<td>0.526</td>
<td>4 hrs 30 mins</td>
<td>16 hrs 15 mins</td>
</tr>
<tr>
<td>St Astier NHL3.5</td>
<td>0.436</td>
<td>4 hrs 25 mins</td>
<td>11 hrs 00 mins</td>
</tr>
<tr>
<td>St Astier NHL5</td>
<td>0.388</td>
<td>4 hrs 40 mins</td>
<td>8 hrs 30 mins</td>
</tr>
<tr>
<td>Jura-Kalk</td>
<td>0.300</td>
<td>3 hrs 05 mins</td>
<td>5 hrs 45 mins</td>
</tr>
<tr>
<td>Bio-E</td>
<td>0.310</td>
<td>3 hrs 25 mins</td>
<td>7 hrs 30 mins</td>
</tr>
<tr>
<td>Chaux de Paviers</td>
<td>0.470</td>
<td>2 hrs 05 mins</td>
<td>6 hrs 00 mins</td>
</tr>
</tbody>
</table>

It can be seen that the initial set is complete within 2-4 hours and shows considerable variation between materials from different manufacturers. The final set had similarly
wide ranging times from 6-16 hours. Some limes set within the limits defined for Portland cement (initial set not earlier than 60 minutes and final set not later than 10 hours), and all of them within approximately 16 hours. These tests were later repeated for setting under water and all the mortars set within 24 hours. It is worth mentioning that the requirement for measuring the time to final set has now been removed from the standards because it is not seen as a useful parameter for site practice. It is however a useful measurement for laboratory investigations. Note also the greatly differing water/lime ratios (column 2 Table 2.1) required to give equal consistency in each case.

In the present study the hydraulic lime used is from the same manufacturer (St Astier) as the above research, therefore the setting time results are of great concern.

2.3.1.3 Water retentivity

Water retentivity is the ability of slurry to retain its mixing water when exposed to absorbent substrate suction. In the case of mortars, water retentivity is an important property to enhance workability (plasticity) and to assure that adequate water is available to hydrate the cementitious components of the mortar [6]. Loss of moisture due to poor water retentivity, in addition to loss of plasticity may greatly reduce the effectiveness of the bond to the masonry units [44]. Insufficient hydration results in a decrease in mortar strength and thus an increase in the sensitivity of the masonry coating to both spalling and cracking [45].

In 2002, Hall and Hoff [7] as stated earlier gave the theoretical basis and quantified the water retaining ability of fresh mortars by a parameter, the desorptivity. Several researchers [5, 46, 47] have determined desorptivity values for cement and lime mortars. Lime-based mortars possess high water retentivity in comparison with cement-based mortars but there is little explanation of the reasons for this. Hendrickx et al. [48] found that the higher the surface area of a binder, the higher the water retentivity of the mortar.

2.3.1.4 The dewatered state

The dewatered state is the state of a wet mix after withdrawal of its water by a dry absorbent before setting. Slurries can be considered as a general case for dewatering and construction mortars are one special category. There is much background theory on the dewatering of industrial slurries and this can be applied to construction materials.
a) Dewatering of slurries

The concept of filterability or dewaterability is the separation of slurry into liquid and filter cake by action of a filter [49]. The dewatering, filtration and sedimentation of slurries is of importance in many industries such as sewage treatment, mineral processing and oil field engineering, in addition to construction. Therefore a large number of studies have been carried out and various tests have evolved. The capillary suction time (CST) method was devised in 1967 by Gale and Baskerville [50] to measure the filterability of sewage sludges. This method measures filterability without using an external source of pressure or suction. The American Petroleum Institute API, developed in 1988 [51], provided a filtration device (which uses pressurised gas) for measuring dewaterability of oil field slurries. The API method has been used in many studies for the filtration of muds and bentonite clay [52-54]. A detailed explanation of all the available test methods for water desorption is given in section 2.4.

Karr and Keinath [55] found that particle size distribution is the property of slurry that most affects its dewaterability.

The theory of water absorption and transport by capillarity in wet slurries is described by the unsaturated Darcy theory. The filtration and sedimentation properties of wet slurries have been described by a similar theoretical model. Philip and Smile [56, 57] have shown that the filtration of a suspension could be described exactly by the same nonlinear diffusion equation as used in unsaturated flow theory. A considerable amount of work based on this theory has been carried out to examine mud and clay filtration in the oil and mineral processing industries [49, 52, 58-60].

b) Dewatering of freshly-mixed hydraulic mortars

The dewatering of construction materials, which is the major concern of this research, has been little studied and there is very little published work on the properties of dewatered wet mixes.

In masonry construction mortars are used in bricklaying, rendering and plastering. In these applications mortars are applied as a wet mix in thin layers to absorbent substrates such as brick, stone and concrete and are dewatered by the capillary suction of these substrates before the beginning of setting. The water desorption of fresh mortar by the substrate depends on both sorptivity of the substrate and desorptivity of the fresh mix [5]. The absorption of the substrate and the water retentivity of the wet mix are in competition and have significant influence on the fresh mortar properties (workability and setting time) and the resultant hardened mortar properties such as adhesion, strength
and durability. If high suction bricks are used with low water-retention mortars this may lead to insufficient hydration of the hydraulic binder and thus a decrease in mechanical strength. The low suction typical of engineering bricks will result in rapid surface saturation and the excess water reduces the bonding capacity of the mortar to the brick causing durability to be reduced. Therefore mortars with low water retention are more suitable for use with low suction bricks and vice versa.

A study by Green et al. [47] showed that the traditional cement mortar of composition 1:3 cement: sand and equivalent mixes containing building lime as a partial substitution for the cement all have similar desorptivities which were much more than a pure 1:3 lime: sand mortar. The study showed also that a water-retaining admixture (methylcellulose-base) can reduce desorptivity to very low levels. Sébaïbi et al. [61] studied the influence of the percentage of various categories of lime on the water retention capacity of CEM1 cement substituted mortar. The results showed that the percentage of cement substituted by lime that yields the minimum water retention depends not only on the type of lime used but on the morphology and chemical nature.

In 2006 Carter et al. [62] showed that cement mortar has very low water retaining ability, which means that it is very water releasing and that hydrated lime mortar is much more water retaining than the equivalent cement mortar. In this work, cement and hydrated lime mortars were used to build two concrete block walls with 10 mm bed joints as shown in figure 2.11.

\[\text{Figure 2.11: Concrete block walls 1.5 hours after construction [62].}\]

Water is abstracted from both sides of the mortar by the concrete blocks. It can be seen that cement mortar is very water releasing, but the hydrated lime mortar is very water retaining. The cement mortar is at one extreme and the hydrated lime mortar at the other extreme of a spectrum of related materials. In-between there are the natural hydraulic
limes (NHLs) and gauged mixes (mixes with an addition of small quantities of pozzolanic materials or any other additives that achieve certain properties) [3].

Collier, et al. [5] experimentally validated the equation $1/A^2 = 1/R^2 + 1/S^2$ between the sorptivity $S$ of the substrate, the desorptivity $R$ of the mortar and the transfer sorptivity $A$ between mortar and substrate for the case of clay brick absorbing water from freshly mixed hydraulic lime and cement mortars. The experimental results showed also that desorptivity varies with both the hydraulicity and the water/binder ratio of the wet mix. Figure 2.12 (upper graph) shows that Portland cement, which has the highest hydraulicity, has a higher desorptivity than NHL3.5 and NHL2. The lower graph shows that for the same NHL a higher water/binder ratio gives higher desorptivity.

![Figure 2.12: Upper graph: cumulative desorbed volume of water per unit area, $i$, plotted against $t^{1/2}$ for (a) OPC mortar; (b) NHL3.5 mortar and (c) NHL2 mortar, all made with water: binder ratio of 0.78: 1. Lower graph: the same data for NHL2 mortars made with water: binder ratios of (d) 1: 1; (e) 0.9: 1 and (f) 0.78: 1 [5].](image)

Pavía et al. [63] tried to correlate bond strength of natural hydraulic lime mortars (NHL5, NHL3.5, NHL2) with water retentivity. Although the water retentivity of the mortars was measured according to BS 4551 (Mortar- methods of test for mortar-chemical analysis and physical testing) [64], which depends on the capillary suction of filter paper and not absorbent brick, for comparison reasons it was satisfactory. The study concluded that NHL mortars have higher water retentivity comparable to those of Portland cement or cement/lime mortars which enables a strong bond with the absorbent brick. The results showed that the bond strength of lime mortars is greatly influenced by water retention followed by water content and finally by strength of the binder.

Patural et al. [65] studied the influence of cellulose ether (CE) which is a water-soluble polymer, on the water retentivity of freshly-mixed cement mortars. Dewatering was
achieved by filter paper according to the method described in DIN 18555-7 [66]. The results showed that the water retaining capacity improved to 92% and 99% with different groups of CE compared to 60% of the non-admixed mortar. They also concluded that the effect of CE on water mobility is with the mortar/unsaturated substrate interface where loss is driven by capillary suction, not by diffusion.

Hendrickx et al. [67] compared the desorptivity of hydrated lime and cement mortars using both methods of capillary suction and pressure filtration. It was found that lime mortar has higher water retentivity in comparison with cement mortar. The authors related this to the particle properties of lime. Lime has smaller particles and a higher specific surface area than cement which leads to a finer pore system resulting in higher suction and lower permeability.

Ince et al. [68] studied the factors affecting the water retentivity of lime and cement mortars in the freshly-mixed state. The results of this study confirm the previous results of [5, 61, 62, 67] that desorptivity depends greatly on the binder hydraulicity. They also found that water retentivity is strongly affected by mix composition. Desorptivity is increased, that is mortar becomes less water retaining, when the water/binder ratio, binder/sand ratio and particle size of sand are increased as shown in figures 2.13, 2.14 and 2.15.

Figure 2.13: Variation in desorptivity with water:binder ratio for CL90 and CEM I mortars of composition 1:2 binder:sand, □ CL90 mortar at 0.5 MPa;▲ CEM I mortar at 0.05 MPa [68].

Figure 2.14: Variation in desorptivity with volume fraction sieved sand contents of zero, 0.5, 1 and 2 for freshly mixed NHL5 with a constant water: binder ratio of 0.78 carried out at pressures of white bar (0.05) MPa;gray bar (0.1) MPa;black bar (0.15) MPa [68].
It can be seen that not a lot of research has been carried out on freshly mixed mortars in the wet state and it is as yet a neglected area of study.

### 2.3.1.5 Effect of dewatering on compressive strength

The mechanical properties of mortars which have been dewatered to varying extents by different absorbent substrates will differ from the same mortars cast in steel moulds. The effect of dewatering by substrate suction on the compressive strength of hydraulic lime mortars was first studied by Carter et al. [62]. Experiments were carried out to measure the 28 and 91 day compressive strengths of mortar from the same mix cast in both steel and brick moulds of the same dimensions as shown in figure 2.16.

Two mortar types were examined: NHL5 and NHL3.5 and the samples were cured in polythene bags at ambient temperatures. The compressive strength of the set dewatered mortars was found to increase by about 31% in the NHL3.5 and by 82% in the NHL5 at 28 days. The increase is higher for NHL5 because it is more hydraulic and hence more
water releasing. This work showed the invalidity of carrying out strength test measurements on a mortar cast in steel mould.

Another recent study by El-Turki et al. [69] was undertaken to examine the influence of dewatering on the compressive strengths of hydrated lime CL90, hydraulic lime and cement mortars. In this study a small open-ended cylindrical mould of 18 mm in diameter and 36 mm in length was used. The freshly mixed mortars were dewatered for 15 minutes by placing the cylindrical moulds on high sorptivity (Leicester Buff) clay facing brick. The results are shown in figure 2.17.

From figure 2.17 it can be seen that the compressive strength of the set dewatered mixes increased with curing time and binder hydraulicity. Since CL90 is the most water retaining binder, the experimental results showed almost no change in compressive strength between the dewatered and non-dewatered samples over time.

![Graphs](image)

Figure 2.17: (a): Compressive strength of ♦, non-dewatered and ■, dewatered CL90 mortars versus time at 14, 28 and 56 days after mixing; (b): compressive strength of ♦, non-dewatered and ■, dewatered CEM I mortars versus time at 14, 28 and 56 days after mixing [69].

However the most water releasing CEM I mortars showed a dramatic increase in compressive strength in the dewatered samples compared with the non-dewatered. Although the results of the above study are similar to those of Carter et al. [62], the dimensions of the designed mould were too small and the method of dewatering at one end by inverting the mould on a brick is not efficient and not representative of practice.

The work of this thesis investigates the effect of dewatering by clay brick using a designed mould of the same standard dimensions of the steel mould (40x40x160 mm) [70]. The work presented here i.e. “in this thesis” shows that the strength and setting time of mortars dewatered via brick mould are not the same as those of non-dewatered samples.
2.3.1.6 Effect of pozzolanic additions

According to Neville [36] Pozzolanic materials are of natural or artificial origin and contain silica, SiO$_2$, in a reactive form. In ASTM C618 [71], a formal definition of pozzolana is “a siliceous or siliceous and aluminous material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ambient temperature to form a compound possessing cementitious properties”. There are many types of industrial and natural pozzolanic waste-products which can be used as binder replacement materials to not only reduce the consumption of production energy, but also contribute to environmental protection [72]. Among these waste-products fly ash (FA), ground granulated blast furnace slag (GGBS) and silica fume (SF) are the most common examples. Addition of these materials to concrete and mortars give improved compressive strength and durability. Recently many attempts to re-use waste glass as a cement or aggregate replacement have been made with some positive results [73-76]. The same pozzolanic materials could also be added to lime mortars. In mortars it is a common practice to add lime to Portland cement in order to improve the early age rheological properties such as water retentivity and workability. However, once setting and hardening has begun, compressive strength becomes the main property required and lime has little role to play because of its low strength. Therefore the replacement of some of the lime with pozzolanic material has been shown to be a great advantage in increasing the strength of lime mortars. Taha et al. [77] examined the effects of fly ash and slag on the bond strength of hydrated lime and Portland cement mortars. The results showed a significant increase in bond strength at 28, 90 and 180 days with 20% substitution of fly ash but no increase was observed with the substitution of slag. Fragata et al. [73] found that glass powder enhances the mechanical properties of hydrated lime or hydraulic lime mortars, but decreases mechanical strength of cement mortars. The authors suggested that this is related to the lack of calcium hydroxide in cement which forces the glass residue to act as an aggregate.

Pozzolanic additives are also suitable for use with lime plasters in the maintenance of historic buildings.

The addition of pozzolanic material to any lime mortar changes its properties: density and strength increase while porosity reduces, depending on the type of pozzolana used. In general the softer pozzolanic materials such as brick dust from clay bricks (fired at less than 950°C) will produce more permeable and flexible mortars; whilst the hard-
burned materials, such as pulverised fuel ash (PFA), will tend to produce a harder mortar [78]. However NO STUDY has been made until the present work on how these materials affect the water retaining ability of freshly mixed hydraulic lime mortars.

2.3.2 Review of water retentivity test methods

The existing methods for testing the water retaining ability of freshly-mixed wet mortars work on one of two principles: capillary suction filtration and pressure filtration.

A- Capillary suction filtration. Desorption is achieved by creating a difference in capillary pressure and measuring the mass of desorbed water after a certain time. This capillary suction can be achieved by the following methods:

1. Contact with absorbent filter paper which is the method used in four standards, three of which are for mortars and one for sludges: BS 4551[64]; BS EN 14701-1[79]; BS EN 413-2 [80] and Belgian standards NBNB14-212 (cited by Hendrickx, 2009) [81]. It is the second of these that relates to sludges.

2. Contact with filter plates which is the method used in two standards: BS EN 459-2 [82] and German standard DIN 18555-7 [66]. Both these standards are for building materials.

B- Pressure filtration. Desorption is achieved by creating a higher pressure on one side of a wet mix or slurry and desorbed water is allowed out at the other side which is at lower pressure. Pressure filtration can be achieved by the following methods:

1. Applying a pressure difference by use of a vacuum pump which is the method adopted by three American standards: ASTM C110 [83]; ASTM C1506 [84] and ASTM C 91[85]. The first standard is for lime mortar and the other two are for hydraulic and masonry cement mortars respectively.

2. Expulsion of water by the action of a pressurised gas which is the method adopted by the American Petroleum Institute (API) pressure cell (1998) [51]. This is used in the oil industry.

3. Expulsion of water by the action of a piston (Faure pressure cell) which is adopted by French standard AFNOR T97-001(cited by Hendrickx, 2009) [81]. This method is common in dewatering of mining sludge and in geotechnics.
The above methods for water retentivity measurement (two of capillary suction filtration and three pressure filtration) are now examined in detail.

2.3.2.1 Suction by filter paper

The European standards [64, 80, 81] all specify the same procedure to determine the water retentivity of mortar. The principle is to subject fresh mortar of standard consistency to suction under defined conditions using filter paper as a substrate. The water retention of a mortar is the mass of water retained as a percentage by mass of the original mix water. The time between the beginning of mixing and the start of the suction period of the water retention test should not exceed 10 minutes. The standard apparatus is shown in figure 2.18.

\[
WR = \frac{(x - (x - \nu)) \times 100}{z}
\]  

(2.60)

where \(z\) is the mass of water present in the mortar before absorption;
\(x\) is the mass of the eight filter papers after absorption;
and \(\nu\) is the mass of the 8 filter papers before absorption.
BS EN 14701-1 [79] specifies another method, the Capillary Suction Time test (CST), which also achieves dewatering by suction applied by the capillary action of an absorbent filter paper. There are some similarities with the BS 4551 [64] and BS 413 [80] tests but no external weight is applied to the sample and filter paper. This method is a frequently used in the filtration measurements of sewage sludges.

The Capillary Suction Time (CST) method was first developed and used in 1967 by Gale and Baskerville [50] and has been frequently applied to quantify the dewatering characteristics of a large number of industrial slurries and suspensions. The CST device provided the first simple, rapid and inexpensive means to quantify the dewatering characteristics of sludges [86]. (Meeten, 1993, 1995 and 1998) [49, 59, 60] carried out a considerable amount of work on the filterability model of the CST test. The principle of the method is to measure the time for a wetting front in a filter paper to travel between two fixed points drawn by the capillary suction pressure of the paper. The device consists of a small stainless steel cylindrical reservoir of 25 mm internal diameter placed on an absorbent filter paper of 185 mm diameter supported by a base as shown in figure 2.19.

The depth of the wet mix in the reservoir is $H$ and the filter paper thickness is $h$. After mixing, the wet mix is immediately poured into the reservoir (to avoid unreliability due to setting) and water begins to be withdrawn by the capillary pressure of the filter paper. The time required for the wet front to cover a circular region between two rings (between $a_o$ and $a$ in figure 2.19) is automatically measured.

![Figure 2.19: Schematic diagram of the capillary suction time apparatus [46].](image)

According to the BS EN14701-1 Standard the reservoir height changes the pressure of the capillary suction therefore a higher reservoir is more suitable for high water
retaining sludges and smaller heights are required for lower water retaining sludges. Two overlapped filter papers are then used for the latter case to increase the absorption capacity. Early work of Meeten et al. [60] showed that the CST is related to the cell radius \( a \) in figure 2.19 and that the CST is proportional to \( a^{-4} \). Meeten et al. [49] describe two cases to determine CST theory depending on the hydraulic permeability of the filter cake or the filter paper. If the CST is measured depending on the permeability of the filter cake then the capillary suction time \( t \) is given by the following equation:

\[
t = \left( \frac{\pi \varepsilon h}{RA_f} \right)^2 \left( a^4 - a_s^4 \right)
\]

where \( \varepsilon \) is the porosity of the filter paper.

\( A_f \) is the cross sectional area of the reservoir.

and \( R \) is the desorptivity of the sample.

From the above equation the desorptivity \( R \) of the sample can be determined from the slope of a plot of \( (a^4 - a_s^4)^{1/2} \) versus \( t^{1/2} \).

Carter et al. [46] used the CST method to find the desorptivity of 1:3 lime/sand mortar and the results showed that \( R \) varies with the reservoir height (10-140 mm) which can be converted to pressure in units of Pa. It was reported also that the maximum practical limit of the pressure of this device is about 16.5 kPa. It should be noted that both methods of suction by filter paper are single point measurement methods and give an indication of the suction of one specific type of substrate that is filter paper and not brick.

Veslind [86] reported that CST is the first simple, rapid and inexpensive method which has been used for more than 20 years in measuring the dewaterability of sludges in waste water and water treatment plants.

### 2.3.2.2 Suction by filter paper plate

The European standards DIN 18555-7:2000 and BS EN 459-2:2010 [66, 82] define the water retaining capacity of freshly mixed mortar as the percentage by mass of water which remains in the mortar after a short time (5 minutes) of capillary suction by absorbent filter paper plates. This is another standard test that relies on the capillary pressure of filter paper. The experimental arrangement is shown in figure 2.20. The apparatus consists of a filter paper plate 190 mm × 190 mm × 2 mm, non-woven tissue 185 mm in diameter, a conical plastic ring, of 140 mm smaller and 150 larger inside diameter, 12 mm in height and two plastic plates of 200 × 200 × 5 mm dimensions. To determine the water retention, a mortar of known consistency is prepared. First the dry
filter plate together with one of the two plastic plates is weighed. Then a piece of nonwoven tissue is placed on the filter plate and the plastic ring, with its smaller opening downwards, on the top and the whole assembly is weighed.

The test should be started 15 s after the completion of the mixing process. The mortar is then poured into the plastic ring as quickly and uniformly as possible and levelled with a straightedge immediately. The assembly is weighed again and covered with the second plastic plate and left to stand for 5 minutes. Finally the test arrangement is inverted and the lower plastic plate removed and weighed with the filter plate. If the mass of the water absorbed by the filter plate exceeds 10 g, the test should be repeated using two or more filter plates.

The water retention WR is calculated using the following equation:

\[
W_1 = \frac{m_8}{m_8 + m_9}
\]

(2.62)

where

- \( w_1 \) is the water fraction of the test mortar;
- \( m_8 \) is the total mass of water in fresh mortar;
- \( m_9 \) is the mass of the dry mortar.

The water content of the mortar in the plastic ring \( w_2 \) is calculated from the following equation:

\[
w_2 = m_{10} \times w_1
\]

(2.63)

\[
m_{10} = m_6 - m_5
\]

(2.64)

where

- \( w_2 \) is the water content of the mortar in the plastic ring;
$m_{10}$ is the mass of mortar in the plastic ring;

$m_5$ is the mass of the plastic plate, filter plate, nonwoven tissue and plastic ring;

$m_6$ is the mass of the plastic plate, filter plate, nonwoven tissue and plastic ring with mortar filling.

The mass of water absorbed by the filter plate $w_3$ is calculated from the following equation:

$$w_3 = m_7 - m_4 \quad (2.65)$$

where

$w_3$ is the mass of water absorbed by the filter plate;

$m_4$ is the mass of the dry filter plate and the plastic plate;

$m_7$ is the mass of the soaked filter plate and the plastic plate.

The relative loss of water from the mortar $w_4$ is calculated from the equation

$$w_4 = \frac{w_3}{w_2} \times 100 \quad (2.66)$$

where $w_4$ is the relative loss of water from the mortar as a percent.

The water retention WR as a percentage is calculated from the equation

$$WR = 100 - w_4 \quad (2.67)$$

The mean of two individual values should be taken to the nearest 0.1%.

Patural et al. [65] used an identical testing method described in German standard DIN 18 555-7:2000. The standardised apparatus is shown in figure 2.21.

![Figure 2.21: DIN 18555-7 test arrangement for filter paper method. 1: two plastic plates; 2: a conical plastic ring; 3: mortar; 4: filter paper; 5: nonwoven tissue [65].](image)

The water retention is calculated by weight differences also but in a more direct and less confusing way. Knowing the initial water content of the mortar under test, the water retention WR is calculated from the following equation:

$$WR = 100 - \frac{w_{fp}}{w_m} \times 100 \quad (2.68)$$
where
\[ w_{fp} \] is the mass of water absorbed by the filter plate.
\[ w_m \] is the water content of the mortar in the plastic ring.

The maximum pressure exerted by the filter paper used in all the above methods is between 10-20 kPa which is much less than that of bricks (100-200) kPa so the tests do not represent the suction experienced by mortar in practice. Again it is a single point measurement.

### 2.3.2.3 Vacuum suction method (ASTM)

Three standards from the American Society for Testing and Materials (ASTM) C110, C91 and C1506 [83-85] describe the specifications for determining the water retention of masonry cement, hydraulic cement and lime mortars respectively. The summary of the technique is as follows. First, the initial flow of the mortar is determined then the mix is subjected to suction through filter paper under controlled vacuum suction for 60 s. Water retention is measured as a percentage of the final flow to the initial flow from a flow table test. The experimental apparatus consists of the flow table device and the filtration assembly. The flow table conforms to specification C230 [87] . The filtration assembly is shown in figure 2.22. This filtration equipment consists of the following components: (1) a perforated dish; (2) a funnel; (3) three-way stopcock; (4) a 1-L flask; (5) filter paper; (6) a rubber gasket; (7) vacuum gauge; (8) vacuum regulator; (9) vacuum pump or aspirator. All the requirements for the dimensions and materials of the constituents are clearly stated in the standard. For example the capacity of the vacuum gauge should be at least 9 kPa in 0.1 kPa increments and the maximum capacity of the vacuum regulator should be 55 kPa, and this is connected to a vacuum pump or water aspirator as shown in figure 2.22.
To determine the water retentivity, first the vacuum should be adjusted to $7 \pm 0.4$ kPa. Then the perforated dish is seated on the greased gasket or greased rim of the funnel. A wetted filter paper is placed in the bottom of the dish. After that, to apply the vacuum to the funnel the stopcock is turned on to check for leaks to ensure the required vacuum is obtained. Then the stopcock should be turned to shut off the vacuum from the funnel. The initial flow of the mortar is determined after mixing by a flow table test. Then the mortar or plaster is returned quickly to the mixing bowl and remixed for 15 s at medium speed. The perforated dish is then filled with mortar to slightly above the rim. The mortar is tamped 15 times with the tamper. The entire filling and tamping should not take more than 2 minutes. The stopcock is turned on to apply the vacuum to the funnel and after suction for 60 s the stopcock is turned off to expose the funnel to atmospheric pressure. A final flow test for the dewatered mortar is determined immediately after suction. The water retention value for the mortar is calculated from the following equation:

$$\text{Water retention, } \% = \left( \frac{A}{B} \right) \times 100 \quad (2.69)$$

Where $A$ is the final flow after suction and $B$ is the initial flow immediately after mixing.
The standardised apparatus of this method was used by Sébaïbi et al. [45], but he uses a different procedure to determine the water retention percentage of lime-cement-sand mortar. The water retention capacity is expressed as a percentage of the initial mixing water mass and is not related to the flow test. The weight of the empty perforated dish with the humidified and drained filter paper was recorded then it was filled with mortar, levelled and weighed again. The difference between the two masses is the mass of the sample placed. Afterwards the apparatus was exposed to vacuum of 7± 0.4 kPa for 15 minutes and the dish reweighed. The water retention capacity is calculated from the following equation:

\[ \text{WR} = \frac{E - e}{E} \times 100 \]  

where

- \( E \) is the mass of the mix-water
- \( e \) is the mass of water lost

The ASTM method and the one adapted by Sébaïbi discussed above are both single point measurements. Atmospheric pressure is about 100 kPa. Therefore these methods which extract water at pressures about 90 kPa, still use pressures that are less than the capillary pressures of bricks.

Hendrickx et al. [48] adapted a version to obtain a measure of water retentivity using several data points. He adapted the set-up of the ASTM C91 standard to contain a larger sample volume. The height of the perforated dish was increased from 20 to 60 mm as shown in figure 2.23. This was done to enable the use of a larger sample and therefore allow the collection of sufficient data points. The test procedure is the same as that followed by Sébaïbi et al. [45].

Figure 2.23: Schematic diagram and image of laboratory set up of the adapted ASTM test apparatus [81].
At the beginning of the test the bottom of the dish is wiped with a dry cloth before it is weighed together with the filter paper and sample. A vacuum pressure is applied to the sample, via the funnel, by turning a valve. In this apparatus the maximum pressure which can be generated by the vacuum pump is about 100 kPa [88]. Measurement of mass is carried out after fixed times of 1, 4, 9, 25, 36, 49, 64 and 81 minutes until the level of pressure cannot be sustained due to air breakthrough occurring. The data points obtained have changed the method from a single point measurement to a multi point measurement which gives sufficient data to plot a graph of the cumulative water loss per unit area, $M$, and the square root of time $t^{1/2}$, since $M = R t^{1/2}$. $R$ here is the desorptivity of the mortar. Figure 2.24 shows typical results of Hendrickx’s adapted ASTM suction test on different mortars at $\Delta p = 20$ kPa applied pressure.

![Figure 2.24: Typical set of results obtained using the adapted ASTM method of Hendrickx suction test on different mortars at 20 kPa applied pressure difference [81].](image)

Figure 2.24 shows the general result that cement mortar (the top curve) is high in desorptivity and hydraulic lime mortars and lime-cement mortars (middle curves) are more water releasing than the hydrated lime mortar (the lower curve). These results may be compared to the work of Collier et al. and Ince et al. [5, 68].

### 2.3.2.4 Pressure filtration by pressurised gas (API pressure cell)

In 1998 the American Petroleum Institute (API) designed a pressure filtration cell for oil-well cement and drilling fluids [51]. Filtration by the API pressure cell has been used for some time in research on dewatering of mining sludges, in geotechnics and in the oil industry. The desorptivity of muds and bentonite clay have been determined by this method in several studies [52-54]. Various studies have used the pressure cell in
desorptivity measurements of cement mortars and grouts [46, 47, 89] and in more recent research on cement- and lime-mortars [5, 68]. This device allows continuous desorption from freshly mixed mortars at a higher range of pressures than all the previous methods. A schematic diagram of the API pressure cell and an image of the laboratory set-up are given in figure 2.25.

![Diagram of API pressure cell](image)

Figure 2.25: (a) Schematic diagram of API pressure cell, (b) the American Petroleum Institute API pressure cell with associated balance and computer.

The principle of this method is to expel water from a wet mix contained in a thick-walled stainless steel cylinder by the action of pressurised nitrogen. The desorbed water from the wet mix is collected from the bottom of the cell in a container placed on a top loading balance. The balance is connected to a computer using software (Sarto Connect) which enables the collection of data (mass of desorbed water) at constant time intervals set by the user. The pressure applied by nitrogen gas used in this method can be higher than 500 kPa, therefore the desorption of water can be measured at a gas pressure equivalent to the capillary suction pressure of an absorbent substrate such as brick. The desorptivity of freshly mixed mortars at a given gas pressure is determined from the slope of the linear part of a plot of the cumulative desorbed volume of water per unit cross sectional area of the cell, \( i \), versus \( t^{1/2} \). A detailed explanation of the modified API pressure cell and experimental arrangement is given in chapter 3.

2.3.2.5 Pressure filtration by piston (Faure pressure cell)

The working principle of this method is the same as for the API pressure cell except that the pressure of compressed air is used to expel water from the wet mortar and is applied
by a piston rather than by pressurised nitrogen gas cylinder. The Faure cell is shown in figure 2.26.

![Schematic diagram and photographic image of the Faure pressure cell apparatus](image)

**Figure 2.26**: Schematic diagram (left) and photographic image (right) of the Faure pressure cell apparatus [81].

The procedure of testing as reported by Hendrickx, [81] is as follows. After the sample is weighed, the piston position is adjusted to allow a volume of sample of approximately 300ml. The wet mix is scooped in three layers on top of the piston with an up and down motion for each layer in order to prevent air inclusion. Then a layer of synthetic tissue is used on top of the sample and the cell closed. To ensure good air-tightness of the pressure cell the sealing surfaces are cleaned carefully, a rubber ring is wetted and fixed in position and the closing clips should be tightened. The final step is to turn the cell upright on a top loading balance and the mass of desorbed water is collected and recorded. The time duration of the test is between 15 and 20 minutes. Hendrickx [81] has reported that the pressure should be increased slowly because any rapid increase causes blockage of the flow at the bottom of the cell. The pressures used in [81] were 100, 200, 300 and 500 kPa. In this method, the desorptivity is also calculated from the linear part of an $i$ versus $t^{1/2}$ plot.

### 2.3.2.6 Evaluation of the methods

The most important parameter of the different methods for dewatering of wet mixes in the case of masonry mortars is the maximum pressure that can be produced. Table 2.2
summarises the maximum pressure that can be achieved of each of the methods described here.

### Table 2.2: Pressure ranges exerted by the test methods described.

<table>
<thead>
<tr>
<th>Method</th>
<th>Typical pressure range (kPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capillary Suction Time (CST)</td>
<td>16.5</td>
</tr>
<tr>
<td>Suction by paper plate</td>
<td>10-20</td>
</tr>
<tr>
<td>ASTM suction test</td>
<td>90-100</td>
</tr>
<tr>
<td>API pressure cell</td>
<td>&gt;500</td>
</tr>
<tr>
<td>Faure pressure cell</td>
<td>500</td>
</tr>
</tbody>
</table>

The standard methods that achieve desorption by the capillary suction of a filter paper, filter plate and CST give an indication of suction of one specific type of substrate that is filter paper. The pressure that can be exerted by filter paper is in the range of 10-20 kPa while the capillary suction pressure of clay brick is between 100-200 kPa. 113 kPa was determined by Ioannou et al. [23] for one specific type of clay facing brick. Carter et al. [46] reported that the pressure exerted in the CST method was 16.5 kPa. Therefore these methods do not test water retention abilities at typical pressures exerted by absorbent substrates such as brick masonry. However the capillary suction methods are simple, rapid and inexpensive and can be used for comparative measurements.

The ASTM methods can exert higher suction pressures (about 100 kPa) but still at the lower end of brick suction pressure. In addition these methods compare mixes of the same flow which is not necessarily a function of water retention.

The API pressure cell method, although not a standard test for masonry mortars, allows a range of applied pressure (> 500 kPa) much higher than the capillary suction of clay brick (100-200 kPa). Therefore desorptivity results obtained by this method are useful because they are made at capillary pressures similar to those of bricks in masonry.

Finally the Faure pressure cell can also achieve high pressure. The highest pressure reported with this method was 500 kPa [67, 81]. These studies found that there are problems such as early air breakthrough at high water content and clogging at the bottom of the cell, especially when testing lime mortar.

The main conclusion of the discussion on methods is that the API is the best available method for measuring the desorptivity of freshly mixed mortars because it can safely apply the pressure which is equivalent to highly absorbent substrates such as brick and block. However the API pressure cell may not achieve full and uniform dewatering of
the mortar sample because gas breakthrough may occur before all the filter cake is completely dewatered.

### 2.3.3 Statistical considerations

The statistical analysis used to investigate the precision and reliability of the API pressure cell is discussed in this section.

The variability between repeated measurements is known as “precision”. It is important to consider precision because the outcome of a measurement cannot be completely controlled due to the presence of unavoidable random errors present in every procedure. The variability of results from a measurement method is due to many different factors including: the operator; the equipment; the calibration of the equipment; the time between measurements and environmental factors such as temperature.

The association between results of repeated measurement is an estimation of the reliability. In experimental research the term “reliability” means “repeatability” or “consistency”. A measurement is considered reliable if it gives the same result when it is repeated. The reliability ratio is the variance of a set of results. Variance is the sum of the squared deviations of a set of results from their mean, divided by the number of results. Standard deviation is a measure of the spread of a set of results, describing how values typically differ from the average of a set. Standard deviation measures the shared variance between measures (it is the square root of the variance).

Variances:

\[ \sigma^2 = \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n} \]  \hspace{1cm} (2.71)

Standard deviation:

\[ \sigma = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n}} \] \hspace{1cm} (2.72)

where \( x_i \) = the individual test result.

\( \bar{x} \) = the average of the test results.

\( n \) = the number of test results.

The International Organization for Standardization (ISO) produced an international standard, BS ISO 5725 [90] consisting of six parts regarding the application of statistical methods for determining the accuracy of measurement methods and results. The Standard considered two conditions of precision, termed repeatability and reproducibility. It defines “repeatability” as “a measure of the closeness of agreement between independent test results obtained with the same method under the same
conditions (same laboratory, operator and apparatus within short intervals of time)” and “reproducibility” as “the closeness of agreement between measurement results obtained with the same method in different conditions (different laboratories, operators and apparatus)”. The measure of precision is often computed as a standard deviation of results. Larger standard deviation reflects less precision. ISO 5725 assumes that the distribution of results is continuous and follows a normal distribution. The confidence limits for both repeatability and reproducibility (standard deviations $\sigma_r$ and $\sigma_R$) are expected to be within a probability of 95%.

The accuracy of measurement methods using repeatability and reproducibility standards have been assessed in many research studies on cement and concrete [91-95]. In this study the repeatability standard deviation (in one laboratory) has been determined for the (API) pressure cell using the above definitions. The data collected was processed using American Society of Testing and Materials (ASTM) E 691 to develop the precision statistics, and the results are reported in chapter 4.

The true value of $\sigma_r$ can only be calculated from a very large number of results. However, Standards enable an estimate, $S_r$ of the standard deviation to be calculated from a smaller number of test results using the following equation:

$$S_r = \sqrt{\sum_i^p s_i^2 / p}$$  \hspace{1cm} (2.73)

where

$S_r$ = the repeatability standard deviation

$S_i$ = the cell standard deviation (cell consists of a number of measurements)

$p$ = number of repeat measurements

The same type of statistical analysis could be applied to the measurement of sorptivity, porosity etc and would give useful information on the measurement errors and the precision with which our experimental results should be reported.

2.4 CONCLUSIONS

- The theoretical considerations of this chapter have shown that Darcy’s law, originally applied to the flow of liquid through saturated porous materials, may be extended to be applicable to capillary flow in unsaturated porous materials (such as construction materials). From this, Sharp Front theory can be applied to solid porous materials to derive the sorptivity and to soft porous materials (pastes and mortars) to
derive the desorptivity and the transfer sorptivity. Sharp Front theory has also been used to derive the time taken to dewater a mortar joint by an absorbent substrate.

- It can be seen that the water retaining ability of freshly-mixed wet mortars and the dewatered state is a neglected area of study. Dewatering of freshly-mixed mortar by absorbent substrates, which is the major focus of this research, has been little studied although it has a great effect on the fresh and hardened properties of mortar.

- This review has shown that desorption is achieved by either capillary suction filtration or pressure filtration. Also it is concluded that the API pressure cell is the best available method for measuring the desorptivity of freshly-mixed mortars because it can safely apply a pressure which is equivalent to highly absorbent substrates such as brick and block.

- The precision of a measurement method is assessed statistically by calculating the repeatability standard deviation (within one laboratory) and the reproducibility standard deviation (within different laboratory).

- It is worth saying that there are a large number of Journal articles on fresh mortars but it is extremely difficult to find published work on dewatered mortars.
3.2 MATERIALS

3.2.1 Clay bricks

Three types of fired clay bricks supplied by Ibstock Brick Limited were used in this study, shown in Figure 3.1. Two are machine-made pressed bricks, (Leicester Buff and Golden Purple). These are facing bricks with quoted high water absorption and low compressive strength as shown in Table 3.1. The other brick (Staffordshire Blue) is a wire-cut extruded brick. It is an engineering brick with quoted low water absorption and high compressive strength (also in Table 3.1). Staffordshire Blue has no perforations. This extruded brick is dark blue-grey in colour and is likely to have been fired at higher temperature than the pressed types, resulting in higher density and much lower porosity. Table 3.1 gives the properties of these bricks as quoted by the manufacturer.
A detailed study of the three types of fired clay brick was carried out to compare sorptivity with the 1 minute initial rate of water absorption according to BS EN 772-11:2011 (Determination of the initial rate of water absorption of clay masonry units) [96], and to compare the vacuum saturation porosity with the 24 h soak water absorption test according to BS EN 772-21:2011 (Methods of test for masonry units: Determination of water absorption of clay and calcium silicate masonry units by cold water absorption) [97] together with satiation porosity. Permeability and capillary pressure were also measured for the two facing bricks, Leicester Buff and Golden Purple, which were both used in the experimental verification of the fundamental equation of water transport in porous materials.
### 3.2.2 Portland cement

Two types of cements were used. The first was Portland cement CEM I manufactured by Hanson Heidelberg cement Ltd. In this type clinker comprises 95-100% of the constituents of the cement and 0-5 % is minor additional constituents (limestone is one possible option). Minor additional constituents are specially selected inorganic natural mineral materials or inorganic mineral materials derived from the clinker production process. The second type of cement used was general purpose Portland-limestone cement CEM II manufactured by Lafarge cement Ltd and designated CEM II/A-LL for which clinker comprises 80-94%. The notation A indicates the range of proportions of the secondary constituent at 6-20% and LL signifies a source of higher purity limestone (\(\text{CaCO}_3 \geq 75\%\) by mass) with low content of total organic carbon (TOC) which, when tested in accordance with pr EN 13639:1999, shall not exceed 0.20 % by mass. Both Portland cements comply with the requirements of BS EN 197-1: 2011 (composition, specifications and conformity criteria for common cements) [98]. The proportion of limestone in the CEM II cement used in this work is 20% as reported by the manufacturer [99]. The chemical composition as oxides (wt %) for the two types of cement are given in Table 3.2.

<table>
<thead>
<tr>
<th>Oxide (wt %)</th>
<th>CEM I</th>
<th>CEM II</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>66.23</td>
<td>61.52</td>
</tr>
<tr>
<td>SiO(_2)</td>
<td>18.85</td>
<td>17.79</td>
</tr>
<tr>
<td>Al(_2)O(_3)</td>
<td>3.61</td>
<td>4.57</td>
</tr>
<tr>
<td>Fe(_2)O(_3)</td>
<td>2.82</td>
<td>2.87</td>
</tr>
<tr>
<td>SO(_3)</td>
<td>3.21</td>
<td>2.87</td>
</tr>
<tr>
<td>MgO</td>
<td>2.02</td>
<td>1.40</td>
</tr>
<tr>
<td>Na(_2)O</td>
<td>0.26</td>
<td>0.11</td>
</tr>
<tr>
<td>K(_2)O</td>
<td>0.70</td>
<td>0.66</td>
</tr>
<tr>
<td>P(_2)O(_5)</td>
<td>0.48</td>
<td>-</td>
</tr>
<tr>
<td>N(_2)O</td>
<td>-</td>
<td>0.55</td>
</tr>
<tr>
<td>Insoluble residue</td>
<td>0.55</td>
<td>-</td>
</tr>
<tr>
<td>Total</td>
<td>99.97</td>
<td>99.98</td>
</tr>
<tr>
<td>free (CaO)</td>
<td>-</td>
<td>1.38</td>
</tr>
<tr>
<td>CaCO(_3) (unburned)</td>
<td>-</td>
<td>17.64</td>
</tr>
</tbody>
</table>

Table 3.2 shows that both cements have significant and nearly similar proportions of the main oxides CaO (lime), SiO\(_2\) (silica), Al\(_2\)O\(_3\) (alumina) and Fe\(_2\)O\(_3\) (iron oxide). The proportions of all other minor oxides are within the limits of the standard [98]. The
percentage of the free lime and unburned limestone in CEM II confirms that the addition of limestone powder is about 20% as reported by the manufacturer.

### 3.2.3 Natural hydraulic limes (NHLs)

The hydraulic limes used in this study were NHL5 supplied by St Astier Lime Group and NHL3.5 and NHL5-Z from Singleton Birch Group UK. The NHL5-Z used in this study is a blend of NHL3.5 and 10% by mass of ground granulated blast furnace slag (GGBS), as reported by the manufacturer. All the hydraulic limes used conform to the requirements of BS EN 459-1:2010 (Building lime: definitions, specifications and conformity criteria) [2]. Table 3.3 gives the physical properties of the NHLs as provided by the manufacturers.

<table>
<thead>
<tr>
<th>Physical &amp; Mechanical Data</th>
<th>St Astier NHL5</th>
<th>Singleton Birch NHL5-Z</th>
<th>Singleton Birch NHL3.5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk density (g/cm³)</td>
<td>0.75</td>
<td>0.59</td>
<td>0.49</td>
</tr>
<tr>
<td>Particle size (passing 90 μm sieve)</td>
<td>93%</td>
<td>99.65%</td>
<td>99.65%</td>
</tr>
<tr>
<td>28 days compressive strength (MPa)</td>
<td>5-9</td>
<td>5-7</td>
<td>3.5-4.5</td>
</tr>
</tbody>
</table>

Table 3.3 shows that the bulk density, particle size (as percent passing 90 μm sieve) and the compressive strength at 28 days (measured on mortars of mix ratio 1:1.3, binder: sand) of NHL5 are all higher than the other limes.

Table 3.4 shows the chemical composition, expressed as oxides, of the NHL5, NHL5-Z and NHL3.5 limes as quoted from the manufacturer’s data.
Table 3.4: Analysis of NHL5, NHL5-Z and NHL3.5 as provided by the manufacturer (Singleton Birch inorganic analysis report, Feb.2009) (St Astier mineralogy and chemistry tests products & raw materials, 2010).

<table>
<thead>
<tr>
<th>Oxide</th>
<th>St Astier NHL5 Wt%</th>
<th>Singleton Birch NHL5-Z Wt%</th>
<th>Singleton Birch NHL3.5 Wt%</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>59</td>
<td>64.91</td>
<td>68.94</td>
</tr>
<tr>
<td>SiO₂</td>
<td>15</td>
<td>9.48</td>
<td>5.96</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>1.92</td>
<td>3.00</td>
<td>1.60</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>0.57</td>
<td>0.91</td>
<td>0.82</td>
</tr>
<tr>
<td>TiO₂</td>
<td>0.18</td>
<td>0.12</td>
<td>0.06</td>
</tr>
<tr>
<td>MgO</td>
<td>1.01</td>
<td>1.35</td>
<td>0.56</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.21</td>
<td>0.31</td>
<td>0.24</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.07</td>
<td>0.08</td>
<td>0.05</td>
</tr>
<tr>
<td>P₂O₅</td>
<td>-</td>
<td>0.14</td>
<td>0.14</td>
</tr>
<tr>
<td>MnO</td>
<td>0.02</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Cr₂O₃</td>
<td>-</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>Mn₃O₄</td>
<td>-</td>
<td>0.25</td>
<td>0.18</td>
</tr>
<tr>
<td>ZrO₂</td>
<td>-</td>
<td>0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>HfO₂</td>
<td>-</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>PbO</td>
<td>-</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>ZnO</td>
<td>-</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>BaO</td>
<td>-</td>
<td>0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>SrO</td>
<td>0.08</td>
<td>-</td>
<td>0.08</td>
</tr>
<tr>
<td>SnO₂</td>
<td>-</td>
<td>&lt; 0.01</td>
<td>&lt; 0.01</td>
</tr>
<tr>
<td>Loss on Ignition at 1025 °C</td>
<td>16</td>
<td>18.89</td>
<td>20.90</td>
</tr>
<tr>
<td>Total</td>
<td>99.58</td>
<td>99.54</td>
<td>99.53</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.41</td>
<td>0.13</td>
<td>&lt; 0.03</td>
</tr>
</tbody>
</table>

From table 3.4 it can be seen that the percentage of the major component CaO is higher in Singleton Birch limes (NHL5-Z and NHL3.5) than in St Astier NHL5 while the SiO₂ percentage is higher in the St Astier NHL5. The silica available to be combined with the CaO determines the hydraulicity of the lime. The St Astier technical data sheet [102] shows that the production of different types of natural hydraulic limes from the same raw material deposits demonstrates that hydraulicity depends on the amount of combined silica. The total amount of silica present consists of soluble silica which is reactive and combinable and the insoluble silica which is inert and un-combinable. The low percentage of clay (as Al₂O₃), sulphur (as SO₃) and alkali (as K₂O and Na₂O)
ensures that the production of these hydraulic limes is based almost totally on the combination of calcium oxide and reactive silica [102].

The percentage of sulphur as SO$_3$ is insignificant as it is much lower than the standard allowed percent which is ≤ 2. The low percentage of SO$_3$ may be introduced by the coal used in the burning of the limestone. It is worth noting that loss on ignition is high in all the limes, St Astier NHL5 has the lowest loss of 16%, although there is no higher limit in BS EN 459-1. The quantities of all the other components are so small that their mineralogical presence is too minute to be relevant.

### 3.2.4 Pozzolanic addition

The pozzolanic material used was ground granulared blast furnace slag (GGBS) from the Civil and Marine Company (now Hanson Heidelberg Cement Group) and conforms to BS EN 15167 (Ground granulated blast furnace slag for use in concrete, mortar and grouts) [103]. GGBS is a waste product in the manufacture of pig iron. It is a pozzolanic material which has cementitious properties. Table 3.5 gives the physical properties and chemical composition, expressed as oxides, of GGBS. The composition shows a significant amount of lime (CaO), active silica (SiO$_2$) and alumina (Al$_2$O$_3$) which are the same oxides present in Portland cement CEM I although the proportions are different. GGBS is chemically reactive and has a large particle size (5-30 μm) as shown in table 3.5.

<table>
<thead>
<tr>
<th>Surface area (m$^2$/kg)</th>
<th>400-600</th>
</tr>
</thead>
<tbody>
<tr>
<td>Particle size (μm)</td>
<td>5-30</td>
</tr>
<tr>
<td>Bulk density (g/cm$^3$)</td>
<td>1.0-1.1</td>
</tr>
<tr>
<td>Colour</td>
<td>Off white</td>
</tr>
<tr>
<td>Oxides</td>
<td>% by mass</td>
</tr>
<tr>
<td>CaO</td>
<td>40</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>37</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>11</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>0.2</td>
</tr>
</tbody>
</table>
3.2.5 Sand
A single source of mortar sand (Croxden) supplied by Carver Ltd and conforming to BS EN 13139 (Aggregates for mortar) [105] was used. After drying in an air oven at 105 °C, it was sieved manually using standard sieves. The (300-600) μm size fraction was used to ensure consistency for all mixes and to eliminate the effect on desorptivity of sand particle size.

3.3 TECHNIQUES USED FOR THE SOLID BULK MATERIALS

3.3.1 Sorptivity measurements

3.3.1.1 Standard sorptivity
A simple procedure for the measurement of sorptivity for bricks and mortars has been described by Gummerson et al. 1980 [28] and Hall and Tse 1986 [106]. For bricks, after drying the sample to constant mass at 105°C in an air oven, the area of the absorbing face is measured to ±1mm with a ruler. The sample is placed on plastic supports in a shallow tray containing water to an immersion depth of approximately 5 mm as shown in figure 3.2. A stop clock is started simultaneously and the sample removed from the water at intervals for weighing. Surface water is dried off with a damp cloth and the sample weighed on a top loading balance measuring to 0.01 g. Each weighing operation is completed as quickly as possible (within 30 seconds) and the clock not stopped during this procedure. The water in the tray is maintained at constant level by adding a little at intervals. The mass of the water absorbed is obtained at minimum of five different times (normally 1, 4, 9, 16, and 25 minutes) and the volume of water absorbed per unit area at each time is calculated. The unit of \( i \) is therefore mm (mm\(^3\)/mm\(^2\)).

Figure 3.2: Arrangement for sorptivity measurement based on [28].

The sorptivity is determined from the gradient of a graph of \( i \) versus the square root of time \( t^{1/2} \) as shown in figure 3.3.
Fig 3.3: Sorptivity $S$ shown equal to the gradient of an $i$ versus $t^{1/2}$ plot.

For the set and hardened mortars used in this work, the sorptivity was measured on rectangular bars of dimensions 40x40x160 mm by the same procedure as used for bricks. The only difference was in drying the mortars to constant mass at 50°C in an air oven instead of 105°C. This temperature was adopted to avoid any chemical alteration or damage to the capillary pore structure of mortars that could have been caused by a higher drying temperature.

3.3.1.2 Measurement of sorptivity by visual inspection of the advance of the wet front

Another method to determine sorptivity is by measuring the distance, $x$, that the water rises into the material by capillarity with time instead of measuring the cumulative absorbed mass. Two ways were used to measure this distance. The first was by drawing lines on the smooth brick face (after removing the frog) at equal distances. The time taken to reach these lines was measured (in minutes) as shown in figure 3.4 (a).

Figure 3.4: (a) First arrangement for visual sorptivity measurement. (b) A diagram showing $S_{vis}$ equal to the gradient of $x$ versus $t^{1/2}$ plot times $f'$. 

\[
S = \text{Gradient mm/min}^{1/2}
\]
The sorptivity equation 3.1 (also 2.39), derived by Hall and Hoff, 2002 [7]:

\[ i = S \frac{t^{\frac{1}{2}}}{f} \]  \hspace{1cm} (3.1)

is divided by the satiation porosity \( f' \) giving

\[ \frac{i}{f'} = S \frac{t^{\frac{1}{2}}}{f'}. \]  \hspace{1cm} (3.2)

Substituting the length of the wetted zone \( x = \frac{i}{f'} \) gives

\[ x = \frac{S}{f'} \frac{t^{\frac{1}{2}}}{f'}. \]  \hspace{1cm} (3.3)

Rearranging equation (3.3) gives

\[ S = \frac{x}{\frac{t^{\frac{1}{2}}}{f'}} \]  \hspace{1cm} (3.4)

In other words

\[ S_{vis} = \text{gradient} \times f' \]  \hspace{1cm} (3.5)

Sorptivity \( S \) was determined from the gradient of a graph of \( x \) versus the square root of time multiplied by the satiation porosity as shown in figure 3.4 (b).

The second way of measuring visual sorptivity was by measuring the distance \( x \) that water reaches at the time intervals used in the standard method (1, 4, 9, 16, and 25 minutes). Lines were drawn every 5 mm on the brick surface along its length as shown in figure 3.5 (a).

![Figure 3.5: (a) Second arrangement for visual sorptivity measurement. (b) A diagram showing \( S \) equal to the gradient of \( x \) versus \( t^{\frac{1}{12}} \) plot times \( f' \) (average of two values).](image)

It was noticed that the water rise was not horizontal and not the same at both sides of the brick, therefore two values of \( x \) were measured: one from the right side and one from the left side of the brick by putting pen marks at each time interval. A precise measure (± 0.2 mm) of distance was made at each time interval.
Sorptivity was determined from the gradient of a graph of $x$ versus the square root of time $t^{1/2}$ as shown in figure 3.5 (b). The average of two values was taken and multiplied by the satiation porosity $f'$ of the brick.

### 3.3.1.3 Vacuum sorptivity

In order to eliminate the resistance of air on the capillary suction of substrates a new method has been developed to measure sorptivity under vacuum. A frame was designed to hold the brick inside a vacuum tank as shown in figure 3.6.

![Figure 3.6: Clay brick held by clamps inside the vacuum tank.](image)

The brick was dried to constant mass at 105 °C before being allowed to cool. The dimensions of the absorbing face were measured. Then the brick was held by a clamp and stand and placed inside a vacuum tank. The chamber and therefore the brick were evacuated and pumping was continued for 30 minutes. The hose of the vacuum tank was then clamped, disconnected from the vacuum pump and the end immersed in water in a second tank. When the clamp was released water was drawn into the vacuum chamber. In vacuum saturation porosity determinations water usually comes from the top of the tank and is stopped when the brick is fully immersed. In vacuum sorptivity an alteration was made to allow water to come to the bottom of the tank and stop when it touched the bottom surface of the brick. The brick had an immersion depth of approximately 5 mm. The hose was clamped again and removed from the water. At the end of the fixed time the hose was released and air allowed to enter the vacuum tank. The brick was weighed and dried again at 105 °C for at least 24 hours to constant mass. As the sorptivity determination needs at least five time intervals, every vacuum sorptivity measurement needed 5-7 days to be completed.

A modification was made to this procedure which was to cool the brick for 3-4 hours after each drying until its temperature was the same as the water temperature. The
reason which drew our attention to this was the higher values of the absorbed volume of water per unit area at the short time intervals of 1, 4 and even 9 minutes. The high temperature of the brick core increased the volume of water absorbed while for longer time intervals of 16 and 25 minutes there was an extra time for the brick to cool in the vacuum tank after the 30 minutes of evacuation.

3.3.2 Initial rate of absorption

The initial rate of absorption test is described in BS EN 772-11:2011 (Methods of test for masonry units: determination of the initial rate of water absorption of clay masonry units) [96]. This standard specifies a method of determining the initial rate of water absorption (IRA) of the bed face for clay masonry units. Its principle is that after drying to constant mass at 105 °C ± 5 °C in an air oven the specimens are allowed to cool to ambient temperature before they were weighed and the dry mass \(m_d\) recorded. The dimensions of the bed face measured and absorbing area \(A_s\) calculated. Then the dry specimen is placed with its bed face supported on small pads so that it is clear of the base of the tray and immersed in water to a depth of 5 mm ± 1 mm for 1 minute. The specimen is then weighed again and the wet mass \(m_s\) recorded. The increase in mass is determined. The initial rate of absorption of clay masonry units, \(I\), is calculated from the following equation [96]:

\[
I = \frac{m_s - m_d}{A_s t} \times 1000
\]

(3.6)

where

- \(m_d\) is the mass of the dry sample (g)
- \(m_s\) is the mass of the wet sample (g)
- \(A_s\) is the area of the absorbing surface (mm²)

and \(t\) is the time (1 minute) in each case.

The units of \(I\) are (Kg m⁻² min⁻¹) as recommended in the standard and, in order to be consistent with the sorptivity units of mm min⁻¹/², the density of water \(\rho\) (1.00 g/cm³) at 4°C was used so that the difference in mass could be converted to volume in mm³. When the time is 1 minute, \(\sqrt{t} = 1\), therefore the units of \(I\) (Kg m⁻² min⁻¹) are consistent with \(S\) (mm/min⁻¹/²).

Therefore the initial rate of absorption is numerically equal to the sorptivity, \(S\), if the straight line graph of \(i\) versus \(t^{1/2}\) passes through the origin.
3.3.3 Porosity measurement

3.3.3.1 Vacuum saturation porosity

This method was used to measure the total accessible open porosity \( f \) as detailed in [8] although it is not a standard test method. In this method a brick was dried to constant mass at 105 °C before being allowed to cool and weighed. The volume was determined, either from measuring the dimensions or by water displacement (using an Archimedes displacement vessel). The sample was then placed in a vacuum chamber which was connected by a hose to a rotary vacuum pump capable of producing a vacuum of \( \approx 30 \) inches of mercury \( (\approx 100 \text{ kPa}) \). The chamber (and therefore the brick) was evacuated and pumping was continued for 30 minutes. The hose was then clamped, disconnected from the vacuum pump and the end immersed in water in a second tank. When the clamp was released, water was drawn into the vacuum chamber. When the sample had become fully immersed, the hose was removed from the water and air allowed to enter the vacuum tank so that atmospheric pressure forces water into the evacuated pores of the bricks. After 24 h of soaking the sample was removed from the water and weighed. The pumping and soaking times (30 min and 24 h) are sufficient according to an early investigation for clay bricks [107] which states that 6 min pumping followed by 15 min soaking gave essentially complete saturation. The mass and hence the volume of water absorbed was determined. The vacuum saturation porosity, \( f \), was then calculated from:

\[
f = \frac{\text{volume of water absorbed}}{\text{volume of sample}} \times 100 \%
\]  

(3.7)

3.3.3.2 Water absorption test (24 hour soak)

The water absorption porosity according to BS EN 772-21:2011 (Methods of test for masonry units: determination of water absorption of clay and calcium silicate masonry units by cold water absorption) [97] is defined as the percentage by mass of pore volume filled in a 24 h soak. The bricks in this test method are dried to constant mass in an air oven at 105°C and allowed to cool to ambient temperature before being weighed and the dry mass \( (m_d) \) recorded. Then they are placed on small pads in a tank of water at room temperature to allow water to be in contact with all faces. The bricks are left submerged for the desired time (24 h and longer times in this study) then taken from the tank and surplus water is removed from their surfaces using a damp cloth. They are weighed and the wet mass \( (m_w) \) is recorded. The water absorption, \( W_m \), is calculated to the nearest 1% using the following equation:
In the work reported here this percentage was calculated at soaking times of 1 min, 9 min, 25 min, 1 h, 24 h and 72 h.

3.3.3.3 Satiation porosity
The satiation porosity or (effective porosity) is determined from one-dimensional capillary absorption using the same simple procedure of sorptivity measurement but with only one weighing needed at the point when the water has reached the top face of the sample. Bricks were dried at 105 °C to constant mass, allowed to cool to ambient temperature, their volume determined before they were weighed and the dry mass recorded. They were placed on their bed faces on small pads in a tank to an immersion depth of approximately 5 mm of water at room temperature to allow water to be absorbed from the bed face only until water reached the top of the brick. They were then taken from the tank, surplus water removed using a damp cloth, weighed and the wet mass was recorded and converted to volume. In this work satiation porosity is denoted by \( f' \) and it is equal to the percentage of the bulk volume filled with water calculated from:

\[
W_m = \frac{m_{w} - m_d}{m_d} \times 100
\]  

(3.8)

3.3.3.4 Satiation porosity under vacuum
In this method the satiation porosity is determined in the same way as the vacuum saturation porosity. The difference is that water was fed into the bottom of a vacuum tank and stopped when the brick bed-face had an immersion depth of approximately 5 mm. Then the hose was clamped and removed from the water. The brick remained under vacuum until the water reached the top of the brick. Then air was allowed to enter the vacuum tank and the brick was removed from the water and weighed. The vacuum saturation porosity which denoted by \( f'' \) was then calculated from equation 3.9. The difficulty of vacuum satiation porosity measurements was the poor visibility through the orange colour of the wall of the vacuum tank (transparent clear tanks do not exist).

3.3.4 Permeability measurement
The saturated permeability of clay bricks was determined using the constant head method according to an extended Darcian flow equation derived by Hall and Hoff [7].
The method is to measure the quantity of water per unit area per unit time that flows unidirectionally through the brick sample under the pressure of a fixed head of water. A direct, simple arrangement was used as shown in figure 3.7.

![Figure 3.7: Test arrangement for permeability measurement.](image)

Cylindrical cores of 20 mm diameter and 21-24 mm in depth were cut from each type of brick perpendicular to the header face, so that the long axis of the core was parallel to bed and stretcher faces. The specimens were dried at 105 °C to constant mass before glued into the ends of short sections of polythene tubes of internal diameter 20 mm. The side surfaces of these specimens were coated with epoxy resin and dried at lower temperature between (50-60) °C to prevent tube melting. When the glue set the specimens were evacuated under vacuum for 30 minutes before being soaked under water.

The polythene tubing was used to provide the constant hydraulic head. The head was maintained at constant level throughout each experiment by adding a little water at intervals. Water was allowed to flow through the brick specimen at each water head height for 90 minutes and was collected in a suitable container which was weighed every 15 minutes. Evaporation losses were reduced by sealing the space between the specimen and the container with cling film.

Four pressure heads of water (1000, 1250, 1500 and 1750 mm) were used. The water collected in this way was used to determine the permeability $K$ using Darcy’s equation [7]:

$$K = \frac{QL}{HA}$$  \hspace{1cm} (3.10)
where

\( Q \) = volume of water flowing through the sample per minute (mm\(^3\)/min).

\( L \) = depth of the sample (mm).

\( H \) = height of water column above the top of the sample (mm).

\( A \) = cross sectional area of the sample (mm\(^2\)).

The volume of water flowing through the sample per minute \( Q \) (mm\(^3\)/min) for each constant hydraulic head was determined from the gradient of a graph of the volume of water collected versus time as shown in figure 3.8 (a). The gradient of a graph of \( Q \) versus the hydraulic head \( H \) (\( Q/H \)), figure 3.8 (b), was used in equation 3.10 to determine the permeability \( K \).

![Figure 3.8: (a) Variation of volume of water collected with time for four hydraulic heads. (b) Variation of \( Q \) with hydraulic heads.](image)

### 3.3.5 Capillary pressure measurement

The equilibrium state of the water in an unsaturated porous material is characterized by its capillary pressure. To measure capillary pressure, rectangular prisms of 160*30*40 mm were cut from each type of brick with the long axis of the prism parallel to the bed and stretcher faces of the brick. After drying the samples to constant mass in an air oven at 105°C, five faces (the four long faces and one small face) were sealed with several layers of epoxy resin which each layer of which was cured for several hours. The initial masses of the bars were recorded and they were then fully immersed in water as shown in figures 3.9 and 3.10.
The main experimental difficulty was to make sure that there was no leakage of air from the sealed faces of the sample until the water absorption stopped. The complete immersion of the sample enabled the detection of any air leaking through the sealed surfaces immediately by the appearance of bubbles. If bubbles appeared, the sample was recovered and recoated with epoxy. The sealed sides ensured that absorption occurred only perpendicular to the inflow face.

The samples were weighed at regular intervals until there was no further change in mass. The volume of absorbed water was determined from the mass increase, giving the volume of pores filled with water. The capillary pressure was determined using the ideal gas equation assuming constant temperature:

\[ p_1 v_1 = p_2 v_2 \]  \hspace{1cm} (3.11)

where

- \( p_1 \) = the pressure of the initial volume of air in the brick before capillary absorption
- and it is equal to the atmospheric pressure (0.1 MPa).
\( v_1 = \text{Initial volume of air (mm}^3\text{)} = \text{vacuum saturation porosity} \times \text{sample volume}. \)

\( p_2 = \text{Final pressure after absorption} = \text{trapped air pressure (MPa)}. \)

\( v_2 = \text{Final volume of air left after capillary rise of water (mm}^3\text{)}. \)

The capillary pressure, which is equivalent to \( p_2 \) in the above equation, is therefore equal to:

\[
p_2 = \frac{p_1 v_1}{v_2}.
\]  

(3.12)

### 3.3.6 Particle size measurement

The particle size of binder materials was measured using a Malvern Mastersizer Micro plus particle size analyzer which uses the technique of laser diffraction. The particle size analysis is based on the principle that all particles scatter light at a range of angles, which is a characteristic of their size. Large particles scatter at small angles and vice versa. The instrument calculates the particle size distribution of a powder; most powders have particles which are of several sizes, forming a distribution.

The particle size distribution of each binder was measured after dispersing the powder in propan-2-ol, with no surfactants, followed by 10 seconds in an ultrasonic bath. The particle size measurement was repeated five times for each material and the mean result was taken.

### 3.3.7 Bulk density measurement

The mass of both binder and sand are required in order to produce mix proportions by volume. Therefore densities of all the binders and the sand were carefully determined. Clean and dry measuring flasks of 50, 100, 250, and 500 cm\(^3\) capacity were weighed to ± 0.01g on a top loading Sartorius CPA2202S balance. They were then filled with untamped material and weighed again. The difference between the two masses was the mass of that volume of binder. This mass was then divided by the volume of the flask and the density for each material was calculated. The mean of four values of density was taken.

### 3.3.8 Flexural and compressive strength measurement

The flexural strength of mortar according to BS EN 196-1 (Methods of testing cement: determination of strength) [108] and BS EN 1015-11 (Methods of test for mortar for masonry: determination of flexural and compressive strength of hardened mortar) [70]
is determined by three-point loading of hardened prism specimens until failure. Flexural strength testing was carried out on six samples of 40x40x160 mm of each mortar mix: three dewatered samples (brick mould) and three non-dewatered (steel mould). First the dimensions (width and depth) of the prisms were measured by a vernier calliper of 100-200 mm with an electronic digital display of accuracy ± 0.03 mm. The samples were then tested in an INSTRON 4507 high resolution digital automatic extensometer of load capacity between 0.5 to 100 kN. This is an electromechanical testing machine which satisfies the accuracy and repeatability errors defined in ISO 7500-1:2004. Each prism was placed with one long face on the supporting rollers with its longitudinal axis normal to the supports as shown in figure 3.11.

All dimensions in mm

![Diagram of prism arrangement](image)

Figure 3.11: Arrangement of loading for determination of flexural strength (BS EN 196-1:2005) [108].

All measurements were carried out at constant strain rate of 0.5 mm/min with automatic recording of load to the nearest ± 0.01 kN and displacement to ± 0.01 mm stored on a computer. The flexural strength was then calculated from the following equation:

\[
R_f = \frac{1.5 F_t l}{b d^2}
\]  

(3.13)

Where \(R_f\) is the flexural strength in MPa; \(F_t\) is the maximum load applied to the middle of the prism at fracture, in N; \(l\) is the distance between the supports (100 mm) and \(b\) and \(d\) are the dimensions of the sides of the section of the prism in mm. Results are presented to \(R_f \pm 0.01\) MPa.

Following flexural failure, each bar produced two samples which were then used for compressive strength measurement. The compressive strength of the mortar was determined on one part of the two pieces resulting from the flexural test for each sample.
The INSTRON 4507 machine used was fitted with stainless steel platens, the bottom being fixed while the top platen was allowed to move. A constant strain rate of 1 mm/min was used for all compressive strength measurements with load and displacement recorded automatically on an attached computer. The compressive strength was then calculated from the following equation:

\[ R_c = \frac{F_c}{bd} \]  

(3.14)

where

\( R_c \) is the compressive strength in MPa; \( F_c \) the maximum load at fracture in N; \( b \) the width of the auxiliary plate (40 mm) and \( d \) the depth of the prism in mm. Results are given to \( R_c \pm 0.01 \) MPa.

### 3.4 TECHNIQUES ON FRESH MORTAR

#### 3.4.1 Desorptivity measurement

The desorptivity, \( R \), of the freshly-mixed mortars was measured using a modified American Petroleum Institute (API) pressure cell [51] shown in figure 3.12. This pressure cell allows a continuous desorption from freshly mixed mortars at a range of pressures as explained earlier in chapter 2.

#### 3.4.1.1 Design of the cell

The pressure cell is made of a thick walled stainless steel cylinder of 54 mm internal diameter and 245 mm height as shown in figure 3.12. The modified version has threaded collars at top and bottom of the cell which is the main difference to the original design of API. These collars make the process of assembly and disassembly of the cell easier and quicker, and also reduce the possibility of pressure loss by contamination of the O-rings and threads with sand grains. The outflow end of the cell, as in the original cell, is fitted with a filter paper of particle size retention less than that of the size of particles of the binder in use; wire gauze mesh; and drain tap.
3.4.1.2 Experimental equipment

Figure 3.13 shows the laboratory arrangement used in desorptivity measurements.

Dry nitrogen gas from a cylinder was used to pressurise the cell. There are two pressure gauges in the system: cylinder pressure gauge A which is used to set the required pressure of each test; and system pressure gauge B which is used to monitor the pressure within the system. There are also two taps in the system. Tap B is closed at the beginning of the experiment to enable the whole system to be pressurised and then is opened to pressurise the cell when the experiment is started. Tap A is used to depressurise the system at the end of the experiment before the pressure cell is removed for cleaning. During an experiment the desorbed water is collected in a container placed on a top loading balance connected to a computer using software (Sarto connect) which enables the collection of data (mass of desorbed water) at constant time intervals set by the user.
3.4.1.3 Mortar preparation

Experimental measurements of desorptivity were carried out on freshly mixed mortars of composition 0.78: 1: 2, water: binder: sand by volume. The mass of both binder and sand required to produce the mix proportions by volume were calculated. To ensure consistency, a constant mixing procedure was followed. An orbital paddle food processor (Hobart Planetary Mixer) was used in mixing the constituents. The required volume of water was added to the bowl first and then the binder material added and mixed at low speed for 1 minute. Next the sand was added and mixing continued for a further two minutes. The mixer was then stopped and, using a plastic scraper, all the mortar adhering to the walls and bottom of the bowl was removed. Mixing was then continued giving a total mixing time of 10 minutes. The mass of each batch was between 1.2 and 1.6 kg which was sufficient to provide 3-4 sequential desorptivity measurements.

3.4.1.4 Measurement procedure

The desorptivity of each freshly mixed wet mortar was measured following the procedure described in [47].

The filter paper used at the base of the cell (figure 3.12) was first saturated with tap water. Any excess water was shaken off before the filter paper was placed on the gauze.
mesh. This filter arrangement was attached to the bottom of the pressure cell by the collar. The bottom collar of the cell was then fixed firmly. Water loss was prevented by closing the drain tap tightly with a spanner.

The pressure in the nitrogen-filled tube shown in Figure 3.13 was set using gauge A before attaching the top collar. This was important in order to prevent any gradual pressure increase in the cell which would cause loss of mix-water from the mortar before the start of the dewatering experiment. The next step is closing both taps A and B in order to avoid nitrogen gas entry into the system. Using this method, when the cell was fully assembled, it could be instantaneously pressurised at a pre-set pressure value and this step thus avoided any mix-water loss before the start of the test.

The pressure cell was then filled carefully from the top with a known mass of wet mortar mix. Care had to be taken that no mortar contaminated the “O” ring sealing surfaces. One third of the mortar mass was loaded at each time and each layer was tamped for approximately 10 seconds in order to remove any trapped air. Following this, the “O” ring was placed in position and the top collar sealed tightly.

Tap B was then opened to allow nitrogen gas at the required pressure to enter the cell. The desorbed water was collected in a container placed on a top loading balance. The mass of the desorbed water was recorded at constant time intervals (5 sec) until gas-breakthrough occurred. The point at which gas breakthrough occurred was easily observed visually as the gas could be seen coming out with the water from the tap. The time to gas breakthrough was noted for each desorptivity measurement before the measurement was concluded. The pressure cell was then depressurised by opening tap A, the filter cake removed and the cell cleaned prior to the next measurement.

Four measurement pressures 0.05, 0.1, 0.15 and 0.2 MPa were used depending on the water retaining ability of the mix being examined. The pressures were determined in a set of trial experiments and were also limited by the scale of the pressure gauge. It was important to carry out desorptivity measurements at appropriate pressures as too high a pressure would result in gas breakthrough occurring before sufficient data could be collected.

The desorptivity, \( R \), at any fixed gas pressure is determined from the slope of the linear part (excluding the data points following gas breakthrough at the end and the discontinuity at the beginning) of a plot of the cumulative desorbed volume of water per unit cross sectional area of the cell, \( i \), versus square root of time \( t^{1/2} \) as shown in figure 3.14.
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3.4.1 Desorption

The departure of the data points from linearity at the end of the linear part of the plot shown in figure 3.14 is due to gas breakthrough and the discontinuity at the beginning of the plot is due to the manner of tamping and the time taken for the collection of the first drops to occur on the top loading balance. Both of these are determined by visual inspection.

3.4.2 Setting time measurement

The setting time of mortars was determined using the Vicat apparatus shown in figure 3.15. The test method is described in BS EN 196-3:2005 + A1:2008 (Methods of testing cement: determination of setting times and soundness) [38].

The principle of the test is to observe the penetration of a needle into the mortar until it reaches a specific value of penetration depth. To determine the initial setting time, a round needle of 1.13 mm diameter which is fixed on a 300 g movable rod was used. After measuring the ambient laboratory temperature by the thermometer supplied with
the apparatus as shown in figure 3.15, a specimen of prepared mortar was placed in the Vicat mould which is 40 mm deep. The penetration depth of the needle, which falls under gravity, was measured. The initial setting time is considered in this work as the time elapsed since the mixing water was added to the binder to when the needle penetration is 4 mm from the base of the mould. The final setting time corresponds to less than 0.5 mm penetration of another needle with a metal attachment hollowed out so as to leave a circular cutting edge of 5 mm in diameter. Setting time was determined for both the dewatered and non dewatered cement and lime mortars at two different ambient temperatures (15°C and 25°C). For the dewatered mortars, the mortar was placed in the brick mould and left for sufficient time to completely dewater. This time to dewater $t_{dw}$ was calculated in advance for each mortar using equation 2.59. Following dewatering, the mortar was removed from the brick mould and then placed into the Vicat mould.

### 3.5 PREPARATION OF FULLY DEWATERED MORTARS

A mould was designed to produce mortar bars of the same dimensions as the standard steel mould of BS EN 1015-part11 [70]. Since the mould was constructed of brick, the wet mortar was fully dewatered prior to setting. Golden purple brick was used for the mould construction as it has relatively high sorptivity. Each brick was cut into two halves along its bed face. The bed face of the brick was used in contact with mortar as would be the case in practice. About 5 mm of the rough brick surface was removed to produce smooth surfaces for casting which were necessary for compressive strength measurement.

Figures 3.16 and 3.17 show the mould consisting of a plastic base plate glued to two bricks of dimension 160x 40 mm (length x depth) and the third dimension left as large as possible. The distance between the brick pieces is 40 mm in order to obtain a mould of size 160*40*40 mm, which corresponds to the standard steel mould (figure 3.18). The ends of the brick pieces were glued to thin plastic plates which could easily be removed. In order to remove the mortar bar from the mould, one of the brick pieces was movable.
Figure 3.16: Photographs of the brick mould. (a) Plan view. (b) Side view.

Figure 3.17: Top view schematic representation of the dewatering by designed brick mould.

Figure 3.18: Typical steel mould of BS EN 1015-11 [70].
3.6 CONCLUSIONS

- This chapter summarizes the materials and experimental techniques used in this study. The materials are traditional, commonly used building materials comprised of different hydraulic binder materials, pozzolanic addition and fired clay bricks. Pressed facing bricks quoted high water absorption and low compressive strength while extruded bricks have higher density and much lower porosity. Portland cements, hydraulic limes and GGBS contain the same oxides but in different proportions.

- Three approaches have been used in this study to investigate the properties of both the fresh and solid states of the materials used. The first approach is to use standard test techniques such as the 24 h soak water absorption, initial rate of absorption and setting time. The second approach is to use non-standard techniques such as measurement of sorptivity, desorptivity and vacuum saturation porosity. The third approach is to develop new techniques such as vacuum sorptivity, visual sorptivity, satiation porosity under vacuum and preparation of fully dewatered mortars by using brick moulds.
CHAPTER 4 INVESTIGATION OF API PRESSURE CELL METHODOLOGY

4.1 INTRODUCTION

The purpose of this chapter is to report a comprehensive investigation into the reliability, applicability, and precision of the American Petroleum Institute (API) pressure cell technique [51] for measuring the water retaining ability (desorptivity) of freshly-mixed mortars. The chapter begins with a brief description of the existing water retentivity test methods which have been explained in detail in chapter 2.

The evaluation of the API pressure cell technique was carried out in two parts: firstly by changing the controlled variables of the experimental set-up which determine the measurement process and affect the results. These include the time interval for the collection of experimental data; the mass (and hence the depth) of mortar placed into the pressure cell; and the pressure of the system. All experimental variables were investigated in this study except temperature. Different measurement time intervals to collect the desorbed water were used at one selected value of applied gas pressure and fixed mass. Different mortar masses and therefore different depths of wet mix were examined at fixed pressure and fixed measurement time intervals. The pressure values investigated were at increasing and at decreasing pressures, keeping the other variables constant. The effect of elapsed time from mixing on desorptivity was also investigated.

Secondly the consequences of the results obtained were evaluated. These include gas breakthrough time and the water content of the resultant filter cake. Gas breakthrough time is one of the consequences of the desorption process and was obtained by recording the time at which gas breakthrough occurred. After placing a fixed amount of fresh mix in the pressure cell, desorbed water is forced out and a cake forms in which the water content is less than that in the original mortar. Eventually nitrogen gas breaks through the cake and desorption of water stops. Gas bubbles are also seen coming out of the tap. The time when this happens is called the gas breakthrough time. It was observed and noted in this study at four different applied gas pressures and its relation with desorptivity and applied pressure are discussed.

SF theory assumes that on complete desorption the water content is uniform along the length of the resultant cake. However it is unlikely that complete desorption was achieved because gas breakthrough happens. Therefore the water contents of the filter...
cakes were determined along their lengths. The uniformity and extent of dewatering was evaluated in this way.

To quantify the variation in the measurements of mass of water desorbed with time, statistical analysis has been used. The International Organization for Standardization (ISO) BS ISO 5725 [90] considered two conditions of precision, termed repeatability and reproducibility. The objective of this part of the chapter is to investigate repeatability and to quantify the variation in measurement results resulting from changing the time interval for data collection, the mass of mortar in the pressure cell and the pressure of the system.

Desorptivity measurements were carried on NHL5 mortar mix of composition 0.78: 1: 2 (water: binder: sand) and this mix was chosen because it is related to other chapters of the present study. Otherwise any other slurry material could be examined in this way.

The above measurements have given the opportunity to examine the errors associated with the pressure cell technique. Although the API pressure cell has been used for desorptivity measurements on mortars for more than 10 years, this is the first time that an examination of the methodology and possible errors has been undertaken.

4.2 EXISTING TEST METHODS FOR WATER DESORPTION

There are several European and American standards which propose methods to test the water retentivity of fresh mortars and slurries in addition to the non-standard method of the American Petroleum Institute (API) pressure cell. All of these tests work on one of two principles: capillary suction filtration or pressure filtration.

Desorption is achieved in most British-European standard methods by capillary suction filtration. The French Faure pressure cell and three American standards adopt pressure filtration methods.

A brief description of these methods is given below.

BS EN 459-2 [82] and German standard DIN 18555-7 [66], are for building materials. These standards work on the principle of capillary suction filtration. The water retention is defined as the percentage by mass of water which remains in the mortar after a short suction time (5 minutes) on filter paper plates.

Another three British standard methods for mortars and one for sludges BS 4551 [64], BS EN 14701-1 [79], BS EN 413-2 [80] and Belgium standard NBNB14-212 (cited by Hendrickx, 2009 [81]) also rely on the capillary suction of a filter paper. They define the water retention of mortar as the mass of water retained after a suction treatment.
under a mass of 2 kg and filter paper sheets as substrate. The result is expressed as a percentage by mass of the original water content.

The capillary suction time (CST) of slurry specified by BS EN 14701-1 relies on the capillary suction of an absorbent filter paper. The rate at which the filter paper becomes wetted is measured and the time necessary for the filtrate to cover the space between two probes, which detect the advance of the wet front on the paper, is measured.

The pressure filtration method is adopted by three ASTM standard test methods C110 [83], C1506 [84] and C91 [85]. These methods measure the ability of mortar to retain water and hence retain consistency. The mix is subjected to a suction of filter paper under applied vacuum pressure on a portion of mortar in a perforated dish. Water retention is measured as a percent of the original consistency from a flow table test.

All the above standard measurements give an indication of the suction of one specific type of substrate: filter paper. The pressure that can be exerted by filter paper is in the range of 10-20 kPa [65, 66] while the capillary suction pressure of clay brick is between 100-200 kPa, and was found to be 113 kPa by Ioannou et al. [23]. Therefore these suction methods do not test water retention abilities at pressures exerted by brick masonry materials.

The American Petroleum Institute (API) pressure cell method, although not a standard test for construction materials, allows a range of applied pressures (> 500 kPa) much higher than the capillary suction of clay brick (100-200 kPa). Therefore desorptivity results obtained by this method are much more useful because they are made at capillary pressures similar to those of bricks in masonry.

4.3 EXPERIMENTAL DETAILS

4.3.1 Desorptivity measurement

Measurements of desorptivity were carried out on freshly-mixed NHL5 mortar of composition 0.78: 1: 2 (water: binder: sand) using the modified API pressure cell technique described in chapter 3. NHL5 was supplied by St Astier Lime Group and conforms to the requirements of BS EN 459- part 1 as stated by the manufacturer. This means that this lime is continuously evaluated on the basis of testing of spot samples according to test methods of EN 459- part 3 (Building lime: conformity evaluation) [109]. The sand used was the 300-600 μm fraction of sieved pre-dried mortar sand supplied by Carver Ltd and conforms to BS EN 13139. Densities of the binder and sand were determined and the mass of both binder and sand required to produce the mix
proportions by volume were calculated. A constant mixing procedure was followed. A detailed description of mortar preparation and desorptivity measurement procedure is given in chapter 3.

4.3.2 Variation of measurement parameters

The evaluation of the American Petroleum Institute (API) pressure cell technique was in two parts. Firstly the variation of measurement parameters was studied. These parameters were time interval of desorbed water collection, mass of mortar in the pressure cell and order of applied gas pressure. Secondly the consequences of parameter variations were examined. These consequences are time to gas breakthrough and the water content and distribution of water in the resultant filter cake.

4.3.2.1 Computer setup for time intervals

In desorption measurements the mass of desorbed water at constant time intervals set by the user was recorded to ± 0.01g on a top loading balance attached to a computer. The software used was a Sarto Connect Windows - 95 /98 /NT based data acquisition program which transfers a series of measured data, from the top loading balance, to any Windows application and stores these data in text fields or tables (e.g. MS Word or MS Excel). The timer interval controls the sequence of values being transmitted to the application. If the time interval is set to 0, the program will transmit the next value without any delay. The maximum value of time interval is 9999 hours, 9999 minutes and 9999 seconds; the minimum value is 0.001 second, the error in time is therefore ± 0.001 second.

An alteration to the computer setup was needed to change the time interval. The input data record structure was changed and a new setting file opened every time to change the data logging time interval.

The values of mass of desorbed water were converted to volumes per unit area \( i \), by dividing by 2290 mm\(^2\) (the cross sectional area of the pressure cell) and values of time \( t \) converted to square root of time. These data were then plotted and desorptivity \( R \) was found from the slope of the linear region of the \( i \) versus \( t^{1/2} \) plot.

To see if the scatter of the plots of cumulative desorbed volume of water per unit area, \( i \), against square root of time could be reduced, five different time intervals were chosen: 1, 2, 3, 5 and 10 seconds at one selected value of applied gas pressure of 0.15 MPa and one selected mass of mortar of 400 g. The measurements were repeated at least once.
The mixes used for repeat time interval measurements were not necessarily from the same batch. This approach can be justified because of the consistent mixing regime used.

### 4.3.2.2 Depth of fresh mix in the pressure cell

Desorptivity measurements were carried out on seven different masses (approximately 150, 200, 300, 400, 500, 600 and 800 g) of NHL5 mortar at 0.15 MPa and time interval of water collection of 5 seconds. In each measurement the required mass of the wet mix was weighed on a two decimal place top loading balance to ± 0.01g and placed into a pre-weighed polythene bag. After the mix was loaded into the pressure cell the polythene bag with the remaining mix was weighed again and the mass remaining was found, thus allowing accurate determination of the mass of mortar in the pressure cell.

After desorption, the depth of the resultant filter cake was measured using a ruler to ±1 mm. Measuring the length of the filter cake was a more accurate procedure than trying to measure the depth of dewatered mortar remaining in the cell.

### 4.3.2.3 Different order of applied gas pressure

Desorptivity measurements are usually performed on a series of 3-4 mortar samples taken from the same batch which are stored in polythene bags to prevent evaporation of the mix water. For the investigation of the effect of the elapsed time between the first sample and the last (which was about 60-75 minutes), desorptivity experiments were carried out at both increasing and decreasing values of pressure on NHL5 mortar of 400 g sample mass and at 5 s time interval for data collection. First increasing values of pressure were examined, thus 0.05 MPa followed by 0.1 MPa, 0.15 and 0.2 MPa. So as time from mixing became greater, the measurement pressure was greater. Then the experiments were repeated with the same pressures in the reverse order on samples from another batch. Therefore there were two mixes measured at each pressure but each with a different elapsed time from mixing.

The consequences of these three experimental parameter variations on desorptivity, gas breakthrough time and water content of the resultant filter cake from the API pressure cell, were examined.
4.3.3 Gas breakthrough time measurement

Gas breakthrough time is one of the consequences of the desorption process. The time in minutes at which gas breakthrough occurred was easily observed visually as gas bubbles can be seen coming out from the tap with the desorbed water. The time was noted for each desorbing pressure. Gas breakthrough can also be seen from the graph of $i$ versus $t^{1/2}$ for each mortar mix as a sudden increase in $i$ value at this point as shown in figure 4.1. Squaring the corresponding time$^{1/2}$ of this point gives the gas breakthrough time.

![Figure 4.1: Typical plot of the cumulative desorbed volume of water per unit area, $i$, against the square root of time showing gas breakthrough.](image)

The gas breakthrough times in minutes of 400 g samples of NHL5 mortar were noted at four different applied gas pressures (0.05, 0.1, 0.15 and 0.2 MPa). A constant time interval of 5s was set for recording the mass of desorbed water.

4.3.4 Water content of the resulting filter cake

The water content as a percent by mass was calculated along the filter cake depth of NHL5 mortar samples of 400 g mass and a 5 s data collection interval desorbed at three gas pressures (0.1, 0.15 and 0.2 MPa). The total length of filter cake was 75 mm ± 1 mm. It was cut into 7 pieces of 10 mm thickness numbering the piece at the tap (outflow) end as 1. Each piece was weighed to the nearest 0.01 g and the mass recorded ($m_w$). The cut pieces were dried to constant mass in an air oven at 105 °C. The pieces were removed from the oven and allowed to cool to room temperature then weighed and the dry mass recorded ($m_d$). The moisture content ($w_c$) percentage was calculated for each piece using the following equation:

$$w_c = \frac{m_w - m_d}{m_w} \times 100$$  \hspace{1cm} (4.1)
The water content of the whole filter cake was also investigated in a separate experiment in the same way by weighing the whole cake, drying in the oven to constant mass at 105 °C and weighing again. The water content of the whole filter cake samples of NHL5 mortar desorbed at 0.1, 0.15 and 0.2 MPa pressure was calculated using the equation 4.1.

For comparison with the water content of a mortar that had been completely dewatered, the water content percent of a mortar bar of dimensions 40*40*160 mm dewatered in the brick mould developed and described in chapter 3 was investigated. The lime mortar specimen was left in the mould for 2 days then de-moulded and was weighed to the nearest 0.01 g and the wet mass recorded ($m_w$). Then it was dried in an oven at 105 °C until constant weight was reached. The mortar bar was removed from the oven and allowed to cool to room temperature then was weighed again and the dry mass recorded ($m_d$). The moisture content ($w_c$) percentage was calculated using equation 4.1. The purpose of this was to determine the extent of dewatering achieved in pressure cell since complete dewatering cannot be achieved because of gas breakthrough. Although it had been assumed in previous work that API pressure cell dewatering was not complete, this has never been quantified.

4.4 RESULTS AND DISCUSSION

4.4.1 Time interval for recording mass of desorbed water

Figure 4.2 shows the variation of the mass of desorbed water with time of 400 g of NHL5 mortar at 0.15 MPa gas pressure for various measurement intervals. For 1s interval three sets of results; for the 2, 3 and 10 s time intervals two sets of results; and for the 5 s time interval six sets of results were recorded.

Each set shows a smooth curve and then a sudden discontinuity at gas breakthrough indicated by a jump in mass. Obviously the number of data points collected at shorter measurement time intervals was much greater than at longer time intervals. At the 10 s time interval too few data points are seen. At the 1s interval there are so many data points that they merge to appear as a line.

Although figure 4.2 shows the general form of the data, the 5 s time interval data were replotted in larger scale for further examination as shown in figure 4.3.

It can be seen that each determination gives results of the same general form but there is some variation of the value of mass with time. This variation is expected and attributed to unavoidable random errors inherent in every test procedure. The variation in mass of
desorbed water largely depends on the manner and skill in which the mortar is loaded and tamped into the pressure cell.

![Desorbed Water Graph](image)

**Figure 4.2:** Variation of the mass of desorbed water with time for NHL5 mortar at measurement time intervals varying from 1 to 10 seconds.
Figure 4.3: Variation of the mass of desorbed water with time for NHL5 mortar at a measurement time interval of 5 s for 6 repeat measurements at 0.15 MPa applied gas pressure.

The correlation between results of repeated measurement is an estimate of the reliability. The statistical method recommended by ASTM standard E691-99 [110] was used to quantify repeatability standard deviation. The 5 s time interval was chosen for the calculation of repeatability standard deviation because there are six measurements at this time interval and although standard E691-99 limits the number of test results to between 2-4 results [110], for practical purposes it was found that a minimum of five results is preferable [111]. The repeatability standard deviation is calculated by the formula given in this standard:

$$S_r = \sqrt{\frac{\sum_i^P S_i^2}{p}}$$

(4.2)

where $S_r$ is the repeatability standard deviation

$S_i$ cell standard deviation of the $i$ measurement interval

$i$ measurement interval

$p$ number of measurement intervals until gas breakthrough

The six results, each at 5 s measurement time, are referred to as a cell and the standard deviation of the cell is $S_i$ at $i$, where $i$ is 5 s interval, until gas breakthrough.

The results of the six determinations at 5 s time interval are shown in table 4.1.
Table 4.1: Variation of mass of desorbed water with time for the six measurements at 5 s time interval. GBT= gas breakthrough.

<table>
<thead>
<tr>
<th>Time (s)</th>
<th>Desorbed water mass (g)</th>
<th>Cell Mean (of 6)</th>
<th>Cell standard deviation</th>
<th>$S_i^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measurement number</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>1.02</td>
<td>0.61</td>
<td>0.67</td>
<td>0.42</td>
</tr>
<tr>
<td></td>
<td>1.02</td>
<td>0.77</td>
<td>1.64</td>
<td>0.80</td>
</tr>
<tr>
<td></td>
<td>1.44</td>
<td>1.73</td>
<td>2.66</td>
<td>0.84</td>
</tr>
<tr>
<td></td>
<td>2.34</td>
<td>2.50</td>
<td>3.47</td>
<td>0.79</td>
</tr>
<tr>
<td></td>
<td>3.08</td>
<td>3.13</td>
<td>3.47</td>
<td>0.79</td>
</tr>
<tr>
<td></td>
<td>4.18</td>
<td>3.86</td>
<td>4.17</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>4.81</td>
<td>4.30</td>
<td>4.77</td>
<td>0.86</td>
</tr>
<tr>
<td></td>
<td>5.33</td>
<td>4.73</td>
<td>5.32</td>
<td>0.99</td>
</tr>
<tr>
<td></td>
<td>5.85</td>
<td>4.73</td>
<td>5.87</td>
<td>1.15</td>
</tr>
<tr>
<td></td>
<td>6.88</td>
<td>5.11</td>
<td>6.56</td>
<td>1.38</td>
</tr>
<tr>
<td></td>
<td>6.62</td>
<td>5.69</td>
<td>7.82</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6.81</td>
<td>6.17</td>
<td>8.30</td>
<td></td>
</tr>
<tr>
<td></td>
<td>GBT</td>
<td>6.68</td>
<td>8.76</td>
<td></td>
</tr>
<tr>
<td>65</td>
<td>GBT</td>
<td>7.12</td>
<td>GBT</td>
<td></td>
</tr>
<tr>
<td>70</td>
<td>GBT</td>
<td>7.56</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 4.1 shows the mass of desorbed water recorded every 5 s for six repeated measurements (the numbered column 1- 6). The time required for gas to break through (GBT) is not the same for all six tests. Test number 3 and 4 took 45 s until gas break through whilst test 5 and 6 took 60 s and test 1 took 70 s. Therefore all six measurements produce desorbed mass data for 45 s.

The standard deviation values of the six results at nine intervals from 5 s to 45 s of the measurement times are calculated and referred to as $S_i$. The sum of the values of $S_i^2$ is 8.08 from which $S_r$ is calculated from equation (4.2).

The repeatability standard deviation $S_r$ for the six measurements is 0.95 g/min as shown below:

$$S_r = \sqrt{\frac{\sum S_i^2}{9}} = 0.947 \approx 0.95 \text{ g/min}$$

The critical value $t_c$ of the $t$ distribution of 9 intervals at 95% confidence level is 2.262 and the confidence limits of the desorbed mass of water is calculated from equation (4.3) [112].

Confidence limits = $\bar{x} \pm t_c S_r / \sqrt{n}$

$$= 3.90 \pm 2.262 \times 0.95 / \sqrt{9}$$

$$= 3.90 \pm 0.96$$
The deviation in the rate of mass of desorbed water is within the confidence limits and this is comparable to the other sets of measurements intervals. The repeatability standard deviation for the set of measurements of time intervals 1, 2, 3 and 10 s are 0.94, 0.16, 0.32 and 0.48 g/min respectively (calculations are given in appendix A). The error in time interval of collecting data was very small as the accuracy in measuring time is 0.001 second.

The data from figure 4.2 (for all the sets of results) were used to calculate the volume of water desorbed per unit area \( i \) and these values were plotted against the square root of time \( t^{1/2} \). The errors in \( i \) will be the same as those in mass. The desorptivity, \( R \), was calculated from the slope of the linear portion of each graph.

In order to calculate the deviation in desorptivity for values with different measurement intervals, the repeatability standard deviation of desorptivity values obtained from the slope of the plot of \( i \) versus \( t^{1/2} \) for measurements at each time interval was also calculated according to standard E691-99 [110]. Table 4.2 shows the desorptivity values with the mean and standard deviation of each set of measurement at each time interval.

### Table 4.2: Desorptivity values of NHL5 mortar measured at 0.15 MPa with different measurement time intervals for collecting the desorbed water.

<table>
<thead>
<tr>
<th>Time interval (s)</th>
<th>Desorptivity (mm/ min(^{1/2}))</th>
<th>Mean</th>
<th>( S_i )</th>
<th>( S_i^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measurement number</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>2</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>1</td>
<td>4.10</td>
<td>3.71</td>
<td>5.51</td>
<td>4.44</td>
</tr>
<tr>
<td>2</td>
<td>5.77</td>
<td>6.55</td>
<td>6.16</td>
<td>5.62</td>
</tr>
<tr>
<td>3</td>
<td>6.04</td>
<td>6.52</td>
<td>5.62</td>
<td>4.62</td>
</tr>
<tr>
<td>5</td>
<td>4.90</td>
<td>4.26</td>
<td>4.81</td>
<td>4.44</td>
</tr>
<tr>
<td>10</td>
<td>5.05</td>
<td>5.19</td>
<td>5.19</td>
<td>5.19</td>
</tr>
</tbody>
</table>

\( \bar{x} = 5.19 \quad \Sigma S_i^2 = 1.62 \)

The repeatability standard deviation \( S_r \) is calculated from equation 4.2 as shown below

\[
S_r = \sqrt{\frac{1.62}{5}} = 0.57 \, \text{mm/} \text{min}^{1/2}.
\]

The confidence limits = \( \bar{x} \pm t_c \, S_r / \sqrt{n} \)

\[
= 5.19 \pm 2.262 \times 0.57 / \sqrt{5}
\]

\[
= 5.19 \pm 0.58
\]

This value of repeatability standard deviation of desorptivity measurements is within the standard repeatability limits at the 95% confidence level (of 0.58). This shows that the variability in desorptivity is acceptable and that the precision of the test method is reasonable.
The average value of the cumulative desorbed volume of water of different number of measurements repeat (for 1 s interval, three; for 2, 3 and 10 s intervals, two; and for the 5 s interval, six) was calculated. A series of graphs of this average volume of water per unit area, \( i \), against the square root of time obtained at each time interval for the 400g of NHL5 mortar at 0.15 MPa pressure are shown in figure 4.4.

It was found that many data points, for example those collected at shorter time intervals, gave a clear linear region. The interval of 10 seconds gave too few data points which were not enough to produce a linear plot.

![Graphs showing variation of cumulative desorbed volume of water per unit area, \( i \), against the square root of time for 400g of NHL5 mortar at 0.15 MPa pressure and different measurement time intervals.](image)

Figure 4.4: Variation of cumulative desorbed volume of water per unit area, \( i \), (average of the sets) with the square root of time for 400g of NHL5 mortar at 0.15 MPa pressure and different measurement time intervals.
4.4.2 Depth of fresh mix in the pressure cell

Desorption measurements were carried out on approximately 150, 200, 300, 400, 500, 600 and 800 g of mortar loaded into the pressure cell. 0.15 MPa pressure and a 5 s data collection interval were used. Each measurement was repeated twice. The resultant filter cake average lengths were approximately 30, 40, 60, 75, 100, 115 and 160 mm respectively.

Figure 4.5 shows the variation of the mass of desorbed water with time for the different masses of mortar used. It can be seen that the small masses of 150 g and 200 g result in only a small number of data points before gas breakthrough: one point for 150 g and two points for 200 g were not enough to produce a plot of \( i \) versus \( t^{1/2} \). The two sets of results are so close, they overlap on the graph and the two points appear as a single point. The number of data points increased for the mix of 300 g but still was not enough to provide a graph.

For masses of 400, 500 and 600 g there were enough data points to produce graphs as seen in figure 4.5.

The 800 g mass was too much and nearly filled the pressure cell whose length is 195 mm (figure 3.12 in chapter 3). This mass took a longer time to desorb and the number of data points was so large that they appear as a line. In addition gas breakthrough did not occur until almost 6 minutes.
Figure 4.5: Variation of the mass of desorbed water with time for different mass of NHL5 mortar at 0.15 MPa gas pressure and 5 s time interval of data collection. The mass of mortar used is shown on the graph in each case.
The average values of the mass of desorbed water of the two measurements from figure 4.5 for 400, 500, 600 and 800 g were used to calculate the desorbed volume of water per unit area and were plotted against the square root of time as shown in figure 4.6.

![Graphs of water desorption](image)

Figure 4.6: Variation of cumulative desorbed volume of water per unit area with the square root of time for NHL5 mortar at 0.15 MPa pressure and 5 seconds time interval of data collection. Mass of mortar used and length of the resultant filter cake are shown on each graph.

Figures 4.6 (a), (b), (c) and (d) all have a clear linear portion with a linear regression factor \((r^2)\) close to 1.

The values of desorptivity, given by the slopes, are given in table 4.3 together with the lengths of the resultant filter cakes. Mean and Standard Deviation (SD) also given.
Table 4.3: Values of desorptivity and lengths of the resultant filter cakes for the range of sample masses examined in the pressure cell.

<table>
<thead>
<tr>
<th>Approximate mass of Mortar in the cell (g)</th>
<th>filter cake length (mm)</th>
<th>Desorptivity (mm/min(^{1/2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Test (1)</td>
<td>Test (2)</td>
</tr>
<tr>
<td>150</td>
<td>28</td>
<td>31</td>
</tr>
<tr>
<td>200</td>
<td>39</td>
<td>41</td>
</tr>
<tr>
<td>300</td>
<td>59</td>
<td>62</td>
</tr>
<tr>
<td>400</td>
<td>75</td>
<td>76</td>
</tr>
<tr>
<td>500</td>
<td>98</td>
<td>103</td>
</tr>
<tr>
<td>600</td>
<td>115</td>
<td>115</td>
</tr>
<tr>
<td>800</td>
<td>160</td>
<td>159</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SD</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

It can be seen from the mean and standard deviation values that the desorptivity results can be given to two decimal places in the rest of this chapter.

### 4.4.3 Order of applied gas pressure

Figure 4.7 shows the variation of the mass of desorbed water with time at four different gas pressures for two sets of measurements (4 samples each) from the same batch of NHL5 mortar. Each sample was of approximately 400 g mass and data were collected at 5 s time intervals. Gas pressures were applied in two orders: increasing and decreasing. First, four samples were examined using increasing values of pressure (0.05 MPa followed by 0.10, 0.15 and 0.2 MPa). The first sample tested at 0.05 MPa was fresh after mixing and the last one, tested at 0.2 MPa, was approximately an hour after mixing. The experiments were then repeated with the same pressures in the reverse order (0.2 MPa followed by 0.15, 0.10 and 0.05 MPa). Therefore there are two values with different elapsed time from mixing at each pressure as shown in figure 4.7. This figure shows that reversing the order from increasing to decreasing resulted in an increase in the mass of desorbed water with time at the high pressure 0.2 MPa but not significantly for the other pressures.
Figure 4.7: Variation of the mass of desorbed water with time of NHL5 mortar at four gas pressures (0.05, 0.1, 0.15 and 0.2 MPa), ◊ increasing pressure value, o decreasing pressure value.

Values of $i$ were plotted against $t^{1/2}$. The values of desorptivities were calculated as before and are shown in a histogram in figure 4.8.

Figure 4.8: Variation of the desorptivity values of NHL5 mortar at four applied gas pressures in two pressure orders (increasing and decreasing). 1-4 denote pressure order.
This figure shows the variation in desorptivity of the NHL5 mortar at both increasing and decreasing pressure. The results show that the desorptivity of the NHL5 mortar decreased after an elapsed time from mixing of an hour when measured at 0.2 MPa. The fourth measurement of the increasing pressure series (no.4) is less than the first measurement of the decreasing pressure (no.1) although both are for the same mortar and at the same pressure. This is because the mortar stiffened during the elapsed time since mixing and became more water retaining.

The maximum desorptivity difference is between the first (after mixing) and last measurement (after an hour). Desorptivity values at pressures 0.1MPa and 0.15 MPa are close for both sets because there is a very short time difference (elapsed time) between the two determinations. The desorptivity at 0.05 MPa pressure was the same in both sets. Therefore the effect of elapsed time of an hour on the water retaining ability of hydraulic limes is not seen at low pressures.

An earlier study by Ince [113] found that one hour from mixing has no significant effect on the desorptivity of NHL5 at 0.05 MPa and the above results confirm this. However, cement mortar desorptivity will be affected to greater extent than NHL5 mortar due to faster stiffening and setting, and this would probably be seen at all measured pressures. Therefore it is concluded that single mixes should be prepared before measurement and that this is better than preparing one large batch on which consecutive measurements are made.

4.4.4 Gas breakthrough time

Figure 4.9 shows the gas breakthrough time in minutes, determined graphically, for NHL5 mortar of 400 g sample mass and 5 s collection time interval, at four applied gas pressures from the graphs shown in figure 4.7. Time is measured to high level of accuracy of ± 0.001 second and pressure is measured to ± 0.01MPa.

Figure 4.9 clearly demonstrates the decrease in gas breakthrough time produced by the increase of applied gas pressure. The higher the pressure the shorter the time is taken for nitrogen to break through the filter cake. For the 0.05 MPa pressure the time taken was more than 2 minutes which was more than double the time taken at 0.1 and 0.15 MPa. This time then dropped to about 40 seconds for 0.2 MPa.
Figure 4.9: Variation of gas breakthrough time with applied gas pressure for NHL5 mortar.

A suitable pressure for any wet mix is one that gives enough time to gather data. However it cannot be taken as a guarantee that the lower the pressures the longer the gas breakthrough time because gas breakthrough time is random and depends on many factors such as the way of loading and tamping the mortar.

The above results confirm the relation between desorptivity and gas breakthrough time by comparison with the results presented later in chapter 5 of this study on hydraulic mortar (Singleton Birch NHL5-Z). This study found that NHL5-Z mortar has a higher desorptivity than NHL5 mortar of the same mix proportions. Gas breakthrough time was found for both mortars at four pressures. Figure 4.10 shows the data for NHL5 mortar already presented in figure 4.9 together with those obtained from NHL5-Z mortar.

Figure 4.10: Gas breakthrough time in minutes for NHL5 and NHL5-Z mortars at four gas pressures.

It can be seen from figure 4.10 that the higher the desorptivity of the material, the longer the time to gas breakthrough except at the highest pressure of 0.2 MPa.
4.4.5 Water content along the filter cake depth

The uniformity and extent of dewatering by the API pressure cell was evaluated by measuring the water content of sections of the resultant filter cake along its length from their wet and dry mass. Figure 4.11 shows the water content percent by mass for 10 mm thick sections of NHL5 filter cakes which had been dewatered at pressures of 0.1, 0.15 and 0.2 MPa.

![Figure 4.11: Variation in the water content of the pressure cell resultant filter cake along its length.](image)

It can be seen that the percent water content is not constant but is lower at the tap (outlet) end. This is to be expected since the filter cake forms at the tap end and grows upwards. Also, the water content at higher pressures is less than from lower pressure. Again this is to be expected as higher pressure will expel more water.

The % water contents in the sections taken from the filter cake dewatered at 0.1 MPa are approximately 14% by mass at the tap end and approximately 16% at the top showing an increase of about 2% along the length of the filter cake.

Figure 4.11 shows the mass percent water content of the original mix (composition 0.78: 1: 2 before dewatering) as a dotted line. The original mix water could never be removed completely in the pressure cell because some of it is used in wetting the constituent particles and in instant hydration.

The water content as mass percent of the dewatered mortar by a brick mould was found to be 8.5%: nearly half that obtained by the pressure cell method. Typical pressure cell experiments therefore produce filter cakes that are not completely dewatered. Dewatering is only about 50% complete by gas breakthrough. The results of figure 4.11 demonstrates clearly that the extent of dewatering by masonry brick is much greater.
than the dewatering by an API pressure cell and for thin layers such as mortar joints this will result in as near complete dewatering as can be achieved.

SF theory assumes that the wetted zone behind the wet front is uniform and has constant water content. This assumption can best be seen if small slices are taken. Constant water content would be found in the completely dewatered sections and the rest of the filter cake would be partially dewatered because of gas breakthrough. Figure 4.12 shows a schematic of the Sharp Front theory approach.

![Figure 4.12: Schematic representation of the sharp front theory approach.](image)

The results of figure 4.11 all indicate a progressive increase in water content with distance from the tap end and show that the water content of the cake is diffuse. This result suggests a limitation of SF theory as applied to wet mixes.

### 4.4.6 Water content of the filter cake as a whole

In a separate set of experiments the water content of the resultant filter cake as a whole was also investigated for 400 g samples of NHL5 mortar desorbed at the same gas pressures of 0.1, 0.15 and 0.2 MPa. The results are given in table 4.4 together with the water content percent of the same mortar dewatered using a brick mould.

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>Water content % (pressure cell)</th>
<th>Water content % (brick mould)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>15.9</td>
<td></td>
</tr>
<tr>
<td>0.15</td>
<td>15.6</td>
<td></td>
</tr>
<tr>
<td>0.20</td>
<td>15.1</td>
<td>8.5</td>
</tr>
</tbody>
</table>
The water content percent of the whole filter cake dewatered at 0.1 was higher than that dewatered at 0.15 or 0.2 MPa as shown in the case of sections. However these results are of limited value in comparison with the water content of the filter cake slices. In comparison with those fully dewatered in a brick mould it shows that samples dewatered in a pressure cell are not suitable for the investigation of the mechanical properties of a set dewatered mix.

4.5 CONCLUSIONS

The summary of results of the work of this chapter is as follows:

- The variations in measurement results generated by changing the measurement time intervals for data collection are small and the standard confidence limits of repeatability standard deviation at 95% confidence interval is 0.58. Therefore the precision of the API pressure cell test is reasonable. A data collection interval of 10 seconds gave too few results to produce a linear portion in the plot of $i$ versus $t^{1/2}$ for mortar.

- There is an optimum mass of mortar mix (400-500g) to be used in the pressure cell which produces an appropriate number of data points for the $i$ versus $t^{1/2}$ plot and which is practical and economic.

- The pressure cell work should be done in one order of applied pressure (either increasing or decreasing) to avoid the changes in desorptivity due to the effect of elapsed time on stiffening of mortar. However it is better just to mix a fresh sample for each test and not one large batch for successive measurements.

- The higher the applied pressure the shorter the time taken for nitrogen gas to break through the wet mortar. Higher pressures should be used with high water retentivity materials in order to gather data within a reasonable time.

- API pressure cell dewatering is not uniform along the length of the resultant filter cake. The water content is lower at the tap end by about 2%.

- Forty to sixty percent of mix water is withdrawn from the wet mix by absorbent brick substrates. API pressure cell dewatering is much less than the complete dewatering by masonry bricks and it is only about 50% complete by gas breakthrough. Therefore dewatering by this way does not produce suitable samples for testing the mechanical properties of set and hardened mortar. This shows a limitation in applying the SF approach to wet mixes and this is largely caused by premature gas breakthrough.
CHAPTER 5  FACTORS AFFECTING THE WATER RETAINING ABILITY OF HYDRAULIC MORTARS

5.1 INTRODUCTION

In masonry construction jointing mortars, renders and plasters are applied in the wet state in thin layers to absorbent substrates such as brick and are dewatered before the setting process begins. The absorption of water from fresh mortar by clay brick depends on both the capillary absorption ability (sorptivity) of the substrate and the water retaining ability (desorptivity) of the fresh mix [5]. The sorptivity of the substrate and other main properties of water transport in fired clay brick are discussed in chapter 6.

The purpose of this chapter is to examine the water retaining ability (desorptivity) of freshly mixed hydraulic lime and cement mortars and to demonstrate the role of many factors: hydraulicity, additive materials, binder particle size and the chemistry of mix water on the water retentivity of these mortars. Knowledge of these parameters will contribute to the ability to control the water retaining ability of a mortar by the incorporation of controlled amounts of additive.

CEM II which is (CEM I +20% limestone powder) was used to investigate the effect of the addition of limestone powder on the desorptivity of Portland cement mortar. Also the effect of a range of volume fractions of the pozzolana ground granulated blast furnace slag (GGBS) added to NHL5 on the desorptivity of mortars made with these binders was examined. Gas breakthrough time was monitored for lime mortars and for lime mortars with a range of GGBS percentage additions. The effects of applied gas pressure on desorptivity are also reported.

The particle size distributions of six binder materials as well as mixes with GGBS were measured and the effects of particle size on desorptivity examined.

The effect of sulphate solution of the same concentration as CEM I extract added as the mix water on desorptivity of hydraulic lime mortar (with and without GGBS) was investigated, with the aim of understanding the role of mix water chemistry on desorptivity.

An American Petroleum Institute API pressure cell using compressed nitrogen gas was used to measure desorptivity.
This is the first time that such work on hydraulic lime and cement mortars has been reported.

5.2 BACKGROUND

The water retaining ability of freshly-mixed wet mortars has been derived and quantified by a well-defined parameter, the desorptivity $R$ [7] as introduced in chapter 2. The water retaining ability of fresh mortars can be measured using an American Petroleum Institute (API) pressure cell which was described in detail in chapter 3. Previous researchers [5, 46, 47, 62] have used this pressure cell technique to measure the desorptivity of cement and lime mortars and have shown that desorptivity depends greatly on the hydraulicity of the binder material used in the mortar mix. Hydraulicity, defined as the ability of the material to set under water, is determined by the amount of clay impurity in the original limestone. The hydraulicity of binders, in order of most to least, is Portland cement, hydraulic limes NHL5>NHL3.5>NHL2. As a result of desorptivity dependence on hydraulicity, hydrated lime has the greatest water retaining ability, hydraulic lime less and Portland cements the least.

It has been shown that lime-based mortars possess high water retentivity in comparison with cement-based mortars, but there is little explanation of the reasons for this. Hendrickx et al. [48] found that the desorptivity of hydraulic mortars was inversely proportional to the specific surface area of the binder. Ince et al. [68] confirmed previous results that desorptivity depends greatly on the hydraulicity of the binder material and also showed that a decrease in sand particle size caused a progressive decrease in desorptivity of mortars in general. This present work investigates for the first time the effect of binder particle size on mortar desorptivity.

The addition of pozzolanic materials such as fly ash, silica fume, glass powder and ground granulated slag as a binder replacement to cement or lime mortars results in increased compressive and bond strength [72-74, 77, 114].

The addition of non-pozzolanic materials such as limestone powder to Portland cement improves the workability and reduces the tendency to bleeding. However the strengths of limestone cement mortars and concretes are lowered due to the less extent of limestone powder participation in the hydration reaction. This reduction in strength is compensated for by the finer grinding of the resulting cement due to easily ground limestone, but the saving of fuel by substituting limestone for some of the clinker is partially affected by the additional energy required for finer grinding. The existing
published work on the use of limestone in Portland cement has an emphasis on amounts of 5% or less [115] although the European standard EN 197-1 [98] allows cement to contain limestone in three different quantity levels. CEM I Portland cement may contain up to 5% minor additional constituents, of which limestone is one possible option. CEM II/A-L and CEM II/B-L, both called “Portland limestone cement,” contain 6-20% and 21-35% ground limestone respectively. However no study has been made until the present work on how these pozzolanic and non-pozzolanic materials affect the water retaining ability of freshly mixed hydraulic mortars.

Ince [113] found that the solution extracted from freshly mixed Portland cement paste made with a water: cement ratio of 1:1 using API pressure cell at 0.5 MPa, when used as mix-water, increases the water releasing ability of CL90 mortar. Even as little as 2% extract solution added to the mix-water was enough to decrease the strong water retaining ability of CL90 pastes and mortars. The chemical analysis of a range of solutions showed that Portland cement extract solution had high sulphate and potassium concentrations. It was also found that the desorptivities of CL90 mortars made using potassium sulphate (K₂SO₄) solution as mix-water were comparable to values obtained using Portland cement extract solution as mix-water.

In this present study the effect of potassium sulphate solution of the same concentration as that obtained from Portland cement paste on NHL5, GGBS and a mix of 50% NHL5+ 50% GGBS mortars was investigated, with the aim of examining the role of sulphate ions in water retentivity.

5.3 EXPERIMENTAL DETAILS

5.3.1 Materials

Materials have already been described in detail in chapter 3. Briefly the hydraulic binders used in this study were two types of Portland cement and three types of natural hydraulic lime. The cements were Ordinary Portland cement CEM I manufactured by Hanson Heidelberg cement Ltd, for which clinker comprises 95-100% of the constituents and 0 to 5 % are minor additional constituents (limestone is one possible option). The second type of cement was general purpose Portland-limestone cement manufactured by Lafarge cement Ltd and designated CEM II/A-LL for which clinker comprises 80-94%, (A) indicates the range of proportions of the secondary constituent at 6-20% and (LL) signifies a source of higher purity limestone with a low content of total organic carbon (TOC). Both Portland cements conform to the requirements of BS
EN 197-1: 2011. The proportion of limestone in CEM II cement used in this work is 20% (as reported by the manufacturer).

The natural hydraulic limes used were NHL5 supplied by St Astier Lime Group, and NHL3.5 and NHL5-Z from Singleton Birch Group UK. The NHL5-Z used in this study is a blend of NHL3.5 and 10% by mass of GGBS, as reported by the manufacturer. All the hydraulic limes used conform to the requirements of BS EN 459-1: 2010.

The pozzolanic material used was ground granulated blast furnace slag (GGBS) supplied by Civil and Marine Ltd, conforming to BS EN 15167-1: 2006.

Sieved mortar sand supplied by Carver Ltd was used, conforming to BS EN 13139: 2002. After drying at 105 °C and sieving, the 300-600 μm size fraction of sand was used to ensure consistency for all mixes and to eliminate the effect on desorptivity of sand particle size.

Table 5.1 summarises the composition expressed as oxides and physical properties of the cement and natural hydraulic lime binders and the GGBS pozzolanic addition.

<table>
<thead>
<tr>
<th>Material</th>
<th>CEM I</th>
<th>CEM II</th>
<th>NHL5</th>
<th>NHL5-Z</th>
<th>NHL3.5</th>
<th>GGBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>66.23</td>
<td>61.52</td>
<td>59</td>
<td>64.91</td>
<td>68.94</td>
<td>40</td>
</tr>
<tr>
<td>SiO₂</td>
<td>18.85</td>
<td>17.79</td>
<td>15</td>
<td>9.48</td>
<td>5.96</td>
<td>37</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>3.61</td>
<td>4.57</td>
<td>1.92</td>
<td>3.0</td>
<td>1.6</td>
<td>11</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>2.82</td>
<td>2.87</td>
<td>0.57</td>
<td>0.91</td>
<td>0.82</td>
<td>0.2</td>
</tr>
<tr>
<td>MgO</td>
<td>2.02</td>
<td>1.4</td>
<td>1.01</td>
<td>1.35</td>
<td>0.56</td>
<td>10</td>
</tr>
<tr>
<td>SO₃</td>
<td>3.21</td>
<td>2.87</td>
<td>0.41</td>
<td>0.13</td>
<td>&lt; 0.03</td>
<td>-</td>
</tr>
<tr>
<td>K₂O</td>
<td>0.70</td>
<td>0.66</td>
<td>0.21</td>
<td>0.31</td>
<td>0.24</td>
<td>-</td>
</tr>
<tr>
<td>Na₂O</td>
<td>0.26</td>
<td>0.11</td>
<td>0.07</td>
<td>0.08</td>
<td>0.05</td>
<td>-</td>
</tr>
<tr>
<td>Ca(OH)₂ (free lime)</td>
<td>-</td>
<td>-</td>
<td>22</td>
<td>21.92</td>
<td>24.16</td>
<td>-</td>
</tr>
<tr>
<td>CaCO₃ (unburned)</td>
<td>-</td>
<td>17.64</td>
<td>23</td>
<td>2.93</td>
<td>1.08</td>
<td>-</td>
</tr>
<tr>
<td>Particle size (μm)</td>
<td>0.5-100</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>5-30</td>
</tr>
<tr>
<td>Bulk Density (g/cm³)</td>
<td>1.12</td>
<td>1.19</td>
<td>0.75</td>
<td>0.59</td>
<td>0.49</td>
<td>1.0-1.1</td>
</tr>
<tr>
<td>Specific surface area (m²/kg)</td>
<td>350</td>
<td>361.38</td>
<td>800</td>
<td>850</td>
<td>900</td>
<td>400-600</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>1.24</td>
<td>7.61</td>
<td>16</td>
<td>18.89</td>
<td>20.9</td>
<td>1.0</td>
</tr>
</tbody>
</table>

In Table 5.1 the oxide composition shows significant amounts of lime (CaO), active silica (SiO₂) and alumina (Al₂O₃) for Portland cement, hydraulic limes and GGBS although the proportions are different. The percent of sulphur as SO₃ is much higher in the cements than the limes. This is to be expected since gypsum is added to cement.
during the grinding stage in manufacture to control setting. The low percentage of SO\textsubscript{3} in limes may be introduced by the coal used in the burning of limestone.

The high quantity of the unburned limestone in CEM II proves that the addition of limestone powder is 20\% as reported by the manufacturer. The quantities of all other minor components are within the limits of the standards.

GGBS has coarser particles (smaller surface area) than the hydraulic limes binders as shown in Table 5.1. It is not currently used in masonry mortars as a binder material, but it is used here to examine the effect of pozzolanic addition and binder particle size on mortar desorptivity. The values of particle size and bulk density of all the binder materials used were determined experimentally and are discussed later.

### 5.3.2 Bulk density measurement

The mass of both binder and sand are required for the mix proportions by volume. Therefore densities of all the binders and the sand were carefully determined as shown in chapter 3. Table 5.2 compares the measured densities with the densities given by the manufacturer.

It can be seen from Table 5.2 that the quoted densities of all the binders and sand were higher than the measured ones by a small amount (not more than 10\%). These differences may be due to the way of handling the material. The bulk density values vary if there is any tamping, vibration or shaking of the container which may settle the particles and give higher mass and as a result higher bulk density.

<table>
<thead>
<tr>
<th>Material</th>
<th>Density values obtained from different volumes of material (g/cm\textsuperscript{3})</th>
<th>Mean density g/cm\textsuperscript{3}</th>
<th>Quoted density g/cm\textsuperscript{3}</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50 cm\textsuperscript{3}</td>
<td>100 cm\textsuperscript{3}</td>
<td>250 cm\textsuperscript{3}</td>
</tr>
<tr>
<td>CEM I</td>
<td>0.90</td>
<td>0.94</td>
<td>0.95</td>
</tr>
<tr>
<td>CEM II/A-LL</td>
<td>1.10</td>
<td>1.04</td>
<td>1.13</td>
</tr>
<tr>
<td>NHL5</td>
<td>0.65</td>
<td>0.69</td>
<td>0.67</td>
</tr>
<tr>
<td>NHL-Z</td>
<td>0.42</td>
<td>0.40</td>
<td>0.44</td>
</tr>
<tr>
<td>NHL3.5</td>
<td>0.45</td>
<td>0.45</td>
<td>0.45</td>
</tr>
<tr>
<td>GGBS</td>
<td>0.98</td>
<td>0.98</td>
<td>0.98</td>
</tr>
<tr>
<td>Sand</td>
<td>1.39</td>
<td>1.42</td>
<td>1.40</td>
</tr>
</tbody>
</table>
5.3.3 Mix design and mortar preparation

All the measurements of desorptivity were carried out on freshly-mixed mortars of composition 0.78: 1: 2 (water: binder: sand) by volume as recommended by the manufacturer to give optimum workability in masonry construction [5, 69] and as used in chapter 4. Several percentages (by volume) of GGBS were added to NHL5 to investigate the effect of this pozzolanic material on the water retentivity of the hydraulic lime mortars. Replacement amounts of 10, 20, 30, 50, 75 and 100% of GGBS were added to NHL5. The details of NHL5 + GGBS mortar mixes are given in table 5.3. The first two columns show the percentage of replacement of NHL5 by GGBS. The remaining columns show the mass of each constituent material in the mix.

Since the density of GGBS is 0.99 g/cm³, the % addition by mass is almost equivalent to the % addition by volume and in the rest of this chapter will simply be referred to as % addition.

Table 5.3: Mix proportions % by volume and masses of constituents of NHL5+GGBS mortars.

<table>
<thead>
<tr>
<th>Mix constituent by volume (%)</th>
<th>Mass of each constituent of the mortar (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NHL5</td>
<td>GGBS</td>
</tr>
<tr>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>90</td>
<td>10</td>
</tr>
<tr>
<td>80</td>
<td>20</td>
</tr>
<tr>
<td>70</td>
<td>30</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td>25</td>
<td>75</td>
</tr>
<tr>
<td>0</td>
<td>100</td>
</tr>
</tbody>
</table>

To ensure consistency, a constant mixing procedure was followed as fully described in section 3.4.1.3 of chapter 3. Briefly the mixing regime is to add the required volume of water to the bowl first, then the binder material, then the additions to the mixer separately before the sand and mixed at low speed for 1 minute. An alternative would have been to mix the binder and addition dry before adding the total to the mixer, but for consistency across all mixes, the mix components were added to the mixer sequentially. Next the sand was added and mixing continued for a further two minutes. Mixing was then continued for a further 7 minutes giving a total mixing time of 10 minutes. The mass of each batch was between 1.2 - 1.6 kg which was sufficient to provide 3 to 4 sequential desorptivity measurements.
5.3.4 Desorptivity measurement

The desorptivity, $R$, of the freshly-mixed mortars was measured using an American Petroleum Institute (API) pressure cell [51]. A full description of the pressure cell method and the experimental procedures of the measurement of desorptivity are given in chapter 3. Briefly the principle of desorptivity measurement is to expel mix water from a freshly mixed mortar contained in a thick-walled stainless steel cylinder by the action of pressurised nitrogen gas at a pressure equal to the capillary pressure of clay bricks. The desorbed water is collected in a container on a top-loading balance connected to a computer using software (Sarto Connect) which enables the collection of data at constant time intervals set by the user.

The desorptivity, $R$, at any fixed gas pressure is determined from the slope of the linear part of a plot of the cumulative desorbed volume of water per unit cross sectional area of the pressure cell, $i$, versus $t^{1/2}$. Four measurement pressures 0.05, 0.1, 0.15 and 0.2 MPa were used.

5.3.5 Sulphate solution preparation

For the investigation of the effect of mix water chemistry on the water retentivity of NHL5 and GGBS mortars, potassium sulphate solution was prepared and used as mix water. A solution of the same sulphate ion concentration (5.38 g/l) as found in CEM1 extract solution [113] was prepared by dissolving 9.75 g of potassium sulphate in 1 litre of deionised water in a volumetric flask using laboratory grade potassium sulphate, which was weighed out on a two decimal place top-loading balance.

5.3.6 Particle size measurement

The particle sizes of nine binder materials: CEM I, CEM II, NHL5, NHL5-Z, NHL3.5, GGBS as well as mixes of NHL5 + 50% GGBS, NHL5 + 75% GGBS and NHL3.5 + 10% GGBS were measured. A Malvern particle size analyzer instrument was used. A detailed explanation of this method is given in section 3.3.5 of chapter 3.
5.4 RESULTS AND DISCUSSION

5.4.1 Effect of binder hydraulicity on desorptivity

Figure 5.1 shows the variation of the cumulative desorbed volume of water per unit area with the square root of time at 4 applied gas pressures for the Portland cement CEM I mortar and two natural hydraulic lime (NHL5 and NHL3.5) mortars and is typical of all the other materials examined in this study.

![Graphs showing cumulative desorbed volume of water per unit area vs. square root of time for CEM I, NHL5, and NHL3.5 mortars at 4 applied gas pressures: 0.05, 0.1, 0.15, and 0.2 MPa. The departure from linearity indicates gas breakthrough.]

Figure 5.1: Plots of cumulative desorbed volume of water per unit area, \( i \), versus the square root of time for CEM I, NHL5 and NHL3.5 mortars at 4 applied gas pressures: 0.05, 0.1, 0.15 and 0.2 MPa. The departure from linearity in each case indicates gas breakthrough.
Gas breakthrough time is the time in minutes to reach the point at which there is rapid and distinct change in \( i \) with time as detailed in chapter 4. Figure 5.1 also clearly shows the gas breakthrough as sudden departure from linearity. It can be seen that the higher the applied pressure, the shorter the time for gas breakthrough to occur as shown earlier in figure 4.9 of chapter 4.

The desorptivity, \( R \), was determined from the slope of the linear part of the graph of \( i \) versus \( i^{1/2} \) for each mortar mix and the results are summarized in table 5.4.

**Table 5.4: Desorptivity values and the applied gas pressures for CEM I, NHL5 and NHL3.5 mortar.**

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>Desorptivity (mm/min(^{1/2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>CEM I</td>
</tr>
<tr>
<td>0.05</td>
<td>3.77</td>
</tr>
<tr>
<td>0.10</td>
<td>4.73</td>
</tr>
<tr>
<td>0.15</td>
<td>5.98</td>
</tr>
<tr>
<td>0.20</td>
<td>7.68</td>
</tr>
</tbody>
</table>

It can be seen that the desorptivity of CEM I mortar which is the most hydraulic is consistently higher than that of St Astier NHL5 and Singleton Birch NHL3.5 (less hydraulic) mortars meaning that CEM I is the less water retaining binder. These results confirm the previous results [5, 46, 47, 62] that lime-based mortars possess high water retentivity in comparison with cement-based mortars. The results of table 5.4 are plotted in figure 5.2.

**Figure 5.2: Variation in desorptivity with applied pressure for CEM I and hydraulic lime NHL3.5 and NHL5 mortars.**
This figure clearly demonstrates that the desorptivity increases with an increase in applied gas pressure, which is equivalent in practical terms to higher suction of the absorbent substrates, for all cement and lime mortars as found previously [68].

The desorptivity of CEM I mortar is higher than NHL5 and NHL3.5 mortars. This is to be expected as CEM I (being PC-based) is the more hydraulic. The St Astier NHL5 mortar has less desorptivity than CEM I by about 40%. However the mortar made with NHL3.5 is seen to be less water retaining than that made with NHL5. Previous results would lead to the expectation that the less hydraulic NHL3.5 would be the more water retaining and give lower values of $R$. All previous work (Collier et al. [5], Green et al. [47], Carter et al. [46, 62] and Ince et al. [68]) has shown that desorptivity, and hence water releasing ability, increases as the hydraulicity of the binder increases. The NHL5 and NHL3.5 are not from the same supplier and clearly it is not possible to compare NHLs in this way unless they are all supplied by the same manufacturer.

5.4.2 Effect of additions on desorptivity

5.4.2.1 Limestone powder addition to CEM I

To show the effect of addition on the desorptivity of hydraulic cement mortar, variation in desorptivity with applied pressure was monitored for two commercial types of Portland cement. One type without addition, Ordinary Portland cement CEM I, and another with limestone addition, Portland limestone cement CEM II, which already contains a controlled level of high-purity limestone powder at 20% (manufacturer’s data sheet). Limestone is at the present time being used in cements produced all over the world. The production of limestone cement leads to reduced levels of CO$_2$ emissions and reduced energy consumption when compared with CEM I production [115]. The main properties of cements that contain additional constituents are influenced by both the particle size distributions and the chemical/mineralogical compositions of the component materials. Limestone acts primarily as an inert additive and often does not participate in the hydration reactions. Therefore the loss in strength of limestone cement is compensated for by finer grinding of the cement. The easier grindability of limestone allows a finer particle size distribution of CEM II and this leads to improved workability and water retentivity of the cement [115].

Figure 5.3 shows that the limestone powder addition reduces the desorptivity of Portland cement or in other words improves its water retentivity by about 20%. This reduction in desorptivity increases at higher pressure (about 25% at 0.2 MPa).
Figure 5.3: Variation in desorptivity with applied pressure for the two types of cement: CEM I and CEM II/A-LL (limestone cement). The CEM I results are also presented in figure 5.2.

Limestone powder is a non-pozzolanic and non-hydraulic material, and therefore it does not have any effects on the chemical composition or the hydraulicity of cement. CEM II consists of 80% CEMI and 20% limestone (CaCO$_3$) which acts as a filler material and therefore the hydraulicity of CEM II is less compared to CEM I. The specific surface area of CEM II is higher due to a well-ground limestone powder and this could have an effect on desorptivity (reduces it) according to [48].

5.4.2.2 GGBS addition to NHL5

Figure 5.4 shows the variation in desorptivity with applied gas pressure for CEM I, NHL5, NHL5-Z mortar and mortar made using GGBS alone as a binder. It can be seen from figure 5.4 that the GGBS has the highest desorptivity at all pressures. The desorptivities of the Singleton Birch NHL5-Z mortar (a blend of NHL3.5 and 10% by mass of GGBS) and CEM I are almost identical, but lower than GGBS. The St Astier NHL5 mortar has the lowest desorptivity and is about 40% less than NHL5-Z and CEM I. The desorptivity of NHL5 mortar is about half that of the mortar made using GGBS alone as the binder.

The inset histogram in figure 5.4 summarises the above data and compares the desorptivity of mortars of the same mix proportion made from six different binder materials and tested at one gas pressure (0.1 MPa).
Figure 5.4: Variation in desorptivity with applied pressure for hydraulic lime, cement and GGBS mortars. Inset: Desorptivity of different binder materials and GGBS used in mortars of the same mix proportions at fixed pressure of 0.1 MPa.

The chemical composition of GGBS shows that GGBS contains significant amounts of lime (CaO) and silica (SiO$_2$) (table 5.1). Therefore it is a true pozzolana and capable of providing siliceous compounds that will chemically react with Ca (OH)$_2$ in the presence of water. It is a self-cementing material to certain extent [116]. The addition of GGBS to hydraulic limes would be expected to increase the hydraulicity of limes and thus increase their desorptivity as a result. Also it has rather a large particle size range (5-30 μm) which is similar to the ranges for Portland cements (table 5.1) and this also may affect the desorptivity. A detailed examination of the effect of particle size is given in the next section.

The purpose of this section is to achieve a detailed investigation of the proportions of GGBS addition required to produce an increase or decrease in desorptivity of hydraulic lime mortars. This is necessary and very important as it will eventually lead to the ability to control the desorptivity of hydraulic lime mortars with additives.

Figure 5.5 summarises the data of table 5.5 and shows the variation in desorptivity of NHL5 mortar with a range of % additions of GGBS at the same four applied gas pressures.
Table 5.5: Desorptivity of NHL5 mortars measured for a range of percentage of GGBS additions at four applied gas pressures.

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>GGBS addition %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0</td>
</tr>
<tr>
<td>0.05</td>
<td>2.81</td>
</tr>
<tr>
<td>0.10</td>
<td>3.73</td>
</tr>
<tr>
<td>0.15</td>
<td>4.44</td>
</tr>
<tr>
<td>0.20</td>
<td>5.44</td>
</tr>
</tbody>
</table>

Figure 5.5: Variation in desorptivity of NHL5 mortars with 0, 10, 20, 30, 50, 75 and 100% GGBS addition at four applied gas pressures.

The results of figure 5.5 show that there is no dramatic effect of GGBS addition on the desorptivity of NHL5 mortar up to 30%. However, increasing the % addition to 50% increases the desorptivity by nearly 25%. There is then a steady increase in desorptivity with higher percentage additions. The results are essentially the same for each pressure. To explain why only the higher percentages of GGBS addition (50%) increase the desorptivity of NHL5 a detailed study of microstructure by Scanning Electron microscopy (SEM) is needed and it is suggested that this is done in future work.

The optimum amount of replacement of GGBS in concrete is about 50% [117, 118] and the British Standard BS 8500-2:2006 (Concrete: specification for constituent materials and concrete) [119] allows the replacement level to be up to 85%. The British Standard BS EN 459-1:2010 for building lime allows pozzolanic or hydraulic materials to be added to building limes up to 20% by mass. The results presented here suggest that the allowed percentage limits of this standard should perhaps be reconsidered and raised.
The NHL5-Z used in this study is a blend of NHL3.5 and 10% by mass of GGBS as stated before. This composition can be reproduced in the laboratory by adding GGBS to NHL3.5. Figure 5.6 compares the desorptivity of NHL3.5 (given in figure 5.2), NHL5-Z (given in figure 5.4) and NHL3.5 + 10% GGBS (mixed in the laboratory and referred to here as “replicate NHL5-Z”).

![Figure 5.6: Desorptivity values of NHL5-Z, NHL3.5 and replicate NHL5-Z (NHL3.5 + 10% GGBS) at four applied gas pressure.](image)

It can be seen that the desorptivities of the NHL5-Z and NHL3.5 + 10% GGBS are almost identical and that both are only marginally higher than the mortar made with NHL3.5 alone.

The results in figure 5.6 demonstrate the effect of GGBS addition on the desorptivity of NHL3.5 which are further supported by the data of figure 5.5 and show that the addition of only 10% GGBS has negligible effect on the desorptivity.

### 5.4.3 Effect of binder particle size on the desorptivity

It has been shown in previous work [68] that coarse sand reduces the water retaining ability of mortars (they become more water releasing). In this study the particle size distributions of different binder materials; CEM I, CEM II, NHL5, NHL5-Z, NHL3.5 and GGBS were measured as shown in figure 5.7.
It can be seen that the finest materials are NHL3.5 (a) and NHL5-Z (b) and then CEM II, GGBS, NHL5 and CEM I, (c), (d), (e) and (f) respectively showing much coarser distributions.

The mean particle size of the finer materials NHL3.5 and NHL5-Z is 4.54 μm and 7.02 μm respectively. CEM II, GGBS, NHL5 and CEM I are coarser with a mean particle size of 12.22, 15.44, 15.64 and 18.21 μm respectively as shown in table 5.6.

Table 5.6: Average particle diameters and distribution percentiles of six binder materials.

<table>
<thead>
<tr>
<th>Binder type</th>
<th>Mean particle size (μm)</th>
<th>Distribution percentiles (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NHL3.5</td>
<td>4.54</td>
<td>90% &lt; 8.03</td>
</tr>
<tr>
<td>NHL5-Z</td>
<td>7.02</td>
<td>90% &lt; 13.24</td>
</tr>
<tr>
<td>CEM II</td>
<td>12.22</td>
<td>90% &lt; 31.20</td>
</tr>
<tr>
<td>GGBS</td>
<td>15.44</td>
<td>90% &lt; 32.64</td>
</tr>
<tr>
<td>NHL5</td>
<td>15.64</td>
<td>90% &lt; 40.01</td>
</tr>
<tr>
<td>CEM I</td>
<td>18.21</td>
<td>90% &lt; 43.16</td>
</tr>
<tr>
<td>NHL3.5+10% GGBS</td>
<td>4.69</td>
<td>90% &lt; 8.39</td>
</tr>
<tr>
<td>NHL5+50% GGBS</td>
<td>15.95</td>
<td>90% &lt; 34.53</td>
</tr>
<tr>
<td>NHL5+75% GGBS</td>
<td>16.42</td>
<td>90% &lt; 37.38</td>
</tr>
</tbody>
</table>

If we compare the desorptivity of the finer particle-size NHL5-Z with NHL3.5 (as shown in figure 5.6) the NHL5-Z mortar has higher desorptivity than that of NHL3.5 mortar and this may be attributed to the larger particle size of the GGBS component in
the NHL5-Z. However NHL5-Z mortar has a higher desorptivity than the coarser NHL5 mortar (figure 5.4). In this case the hydraulicity must be the controlling factor. For CEM I and CEM II, it is clear that the fine limestone powder addition reduces the mean particle size of CEM II to 12.22 μm (table 5.6). In the case of GGBS and NHL5, since the particle size is nearly the same for each, it must also be the hydraulicity which accounts for the difference in desorptivity.

It is obvious from these results that the binder particle size has a contradictory effect on desorptivity. Therefore the high desorptivity of GGBS mortar is most likely to be due to the high hydraulicity of the GGBS addition and the particle size may have little effect.

The average particle diameter of NHL3.5 is (4.54 μm). When mixed with GGBS, the average particle size of the resulting replicate is nearly the same of that of NHL3.5 alone (4.69 μm) (7th row of table 5.6).

The particle size distributions of the “replicate” NHL5-Z (NHL3.5 + 10% GGBS) and the commercial NHL5-Z are compared with NHL3.5 in figure 5.8. This figure shows the similarity in particle size distribution of the commercial NHL5-Z and replicate NHL5-Z (NHL3.5 + 10% GGBS). Since both NHL5-Zs have the same desorptivity (as shown in figure 5.6), it confirms that NHL5-Z can be replicated in the laboratory for research purposes.

![Figure 5.8: Particle size distributions of NHL3.5, NHL 3.5 with the addition of 10% GGBS, and NHL5-Z.](image-url)
NHL5 has nearly the same mean particle size as GGBS but about half the desorptivity of a mortar made using GGBS alone as a binder as shown in figure 5.4.

The particle size distributions of NHL5, NHL5 + 50% GGBS, NHL5 + 75% and GGBS are compared in figure 5.9.

![Particle Size Distributions](image)

**Figure 5.9: Particle size distributions of NHL 5, NHL5 plus 50 and 75% GGBS and GGBS.**

This figure shows that the particle size distribution of NHL5 is not greatly affected by the addition of the higher percentages of GGBS. The average particle size is increased from 15.64 to 15.95 μm and to 16.42 μm with 50% and 75% GGBS addition respectively (as shown in the last two rows of table 5.7). However there is a significant increase in desorptivity of NHL5 mortar with 50% and 75% addition as shown in column 6 and 7 of table 5.5.

These results confirm that the binder particle size has little effect on desorptivity comparing to the hydraulicity which is the major factor.

It is worth noting that the fineness values of the cements, limes and GGBS provided by the manufacturers (table 5.1) are nearly the same as the values determined experimentally.

### 5.4.4 Effect of mix water chemistry on desorptivity

The work reported in previous sections has shown that the water retaining properties of hydraulic lime mortars decreases as their hydraulicity increases. This section
investigates this effect further. In this section the effect of mix water chemistry on desorptivity is examined.

Green et al. [47] found that the addition of a small amount of cement to lime greatly increases the desorptivity of lime mortar. They found that the replacement of 30% of lime binder by cement had almost doubled the desorptivity of the 1:3 (lime: sand) mix at 0.5 MPa. Ince, 2009 [113] found that even 2% of Portland cement extract solution added to the mix water was enough to decrease the strong water retaining ability of CL90 pastes and mortars. The study analysed a range of binder materials extract solutions and found that potassium and sulphur (as sulphate) are present in high concentration in Portland cement, less in hydraulic limes and exist only as impurities in the hydrated lime CL90. It was found that the desorptivity values of CL90 mortars made using potassium sulphate (K₂SO₄) solution as mix water were very similar to those obtained by using CEM I extract solution as mix water.

In this work a sulphate solution was used as the mix water to simulate the effect on desorptivity of adding CEM I to hydraulic lime and GGBS mortars. Potassium sulphate solution was prepared of the same concentration as CEM I extract by dissolving 9.75 g of K₂SO₄ in 1 litre of water [113].

Table 5.7 compares the desorptivities of NHL5, GGBS and a mix of 50% NHL5 + 50% GGBS mortars made with both water and K₂SO₄ solution as mix-water over a range of applied gas pressures. It is very clear that the K₂SO₄ solution increases the desorptivity not only for NHL5 mortar (columns 1 and 2) but also for the GGBS mortar (columns 5 and 6) at all pressures by an amount that depends on the gas pressure used.

Table 5.7: Desorptivity of a range of NHL5 and GGBS mortars with both water and sulphate solution as mix-water. Amounts in brackets are the increase in desorptivity with sulphate solution compared to water.

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>NHL5/ water</th>
<th>NHL5/ K₂SO₄ solution</th>
<th>NHL5+ 50% GGBS/ water</th>
<th>NHL5+ 50% GGBS/ K₂SO₄ solution</th>
<th>GGBS/ water</th>
<th>GGBS/ K₂SO₄ solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>2.81</td>
<td>3.32 (18% ↑)</td>
<td>3.61</td>
<td>3.98 (10% ↑)</td>
<td>5.41</td>
<td>5.73 (6% ↑)</td>
</tr>
<tr>
<td>0.10</td>
<td>3.73</td>
<td>4.25 (14% ↑)</td>
<td>4.31</td>
<td>5.06 (17% ↑)</td>
<td>6.38</td>
<td>6.89 (8% ↑)</td>
</tr>
<tr>
<td>0.15</td>
<td>4.44</td>
<td>4.82 (9% ↑)</td>
<td>5.05</td>
<td>6.42 (27% ↑)</td>
<td>6.98</td>
<td>7.60 (9% ↑)</td>
</tr>
<tr>
<td>0.20</td>
<td>5.44</td>
<td>5.83 (7% ↑)</td>
<td>6.51</td>
<td>7.64 (17% ↑)</td>
<td>7.97</td>
<td>8.84 (11% ↑)</td>
</tr>
</tbody>
</table>
Figure 5.10 presents the NHL5 and GGBS mortar results graphically which makes it clear that the effect of sulphate ions is of similar magnitude on each mortar.

These results suggest that sulphate ions are the main factor that is responsible for increasing desorptivity. The higher concentration of sulphate in Portland cement and hydraulic limes is consistent with the fact that the desorptivity of these binders are higher than that of hydrated lime.

The mechanisms of this are not yet fully understood. It is likely that the strong –ve charge of sulphate causes an electrostatic effect as seen in the dispersion of clays, silts and other fine particles (e.g. in river estuaries).

5.4.5 Effect of GGBS addition on gas breakthrough time

It has been shown earlier in chapter 4 that the higher the pressure the shorter the time taken for nitrogen to break through the filter cake; and that the higher the desorptivity of the material, the longer the time to gas breakthrough. In this chapter the effect of GGBS addition on gas breakthrough time of NHL5 mortar at fixed applied gas pressure of 0.1 MPa was measured for a range of percentages added. Results are shown in figure 5.11. At a fixed pressure, the gas breakthrough time of NHL5 mortars increased as the amount of GGBS increased, as shown in figure 5.11 (a). The gas breakthrough time increased in proportion to desorptivity: the higher the desorptivity (results from row 2 of table 5.5) of the mortar, the longer the time for the gas to breakthrough as shown in figure 5.11 (b).
This is as an extra observation for the necessity of selecting a suitable pressure for measuring desorptivity of any hydraulic material which gives enough time to gather data before gas breakthrough.

In general it would not be expected that gas breakthrough time can be related to any property or to be predictable since it largely depends on the manner in which the mortar is loaded and tamped into the pressure cell.

Figure 5.11: Variation of gas breakthrough time (GBT) of NHL5 mortar at fixed gas pressure with (a) percentage of GGBS addition and (b) desorptivity.

### 5.5 CONCLUSIONS

The results given in this chapter show that:

- Lime-based mortars possess high water retentivity in comparison with cement-based mortars. This finding applies at all desorbing pressures. But when comparing the water retaining abilities of hydraulic limes it is important that they all come from the same supplier.
• In general desorptivity increases with an increase in applied gas pressure and the higher the applied pressure, the shorter the time for gas breakthrough to occur for all cement and lime mortars.

• It is possible to examine the effect of addition materials on the water retaining behaviour of freshly mixed hydraulic mortars in a quantitative way by measuring the desorptivity.

• The limestone powder addition is an inert filler which slightly reduces the desorptivity of Portland cement. In contrast the pozzolanic addition GGBS, which is widely used in concretes, increases the hydraulicity of lime mortars and consequently increases the desorptivity. GGBS additions to NHL5 up to 30% have little measurable effect in terms of the water retaining ability of freshly mixed hydraulic lime and cement mortars. However increasing the percent addition to 50% increases the desorptivity by nearly 25%. The British Standard BS EN 459-1:2010 is at present allows percentage addition up to 20% of GGBS and this could be raised.

• It has been possible to confirm that the water retaining properties of an NHL5-Z mortar may be replicated in the laboratory by combining the separate constituents NHL3.5 and GGBS and comparing the desorptivity and the particle size distributions with those of NHL5-Z mortar of the same mix proportions.

• At a fixed pressure, the gas breakthrough time of NHL5 mortars was increased as the amount of GGBS addition was increased.

• It has been shown previously that coarser sand decreases the water retaining ability of mortars. The results of this study indicate that the particle size distribution of the binder material has little effect on the desorptivity compared to hydraulicity and that hydraulicity of the binder may considered as a contributing factor to the water retaining ability of mortars in general.

• Adding sulphate ions to the mix-water of hydraulic lime mortars reduces their water retaining ability, as has been previously found for hydrated lime mortars. Sulphate also reduces the desorptivity of mortars made with GGBS as binder. It may be suggested that the strong negative charge of sulphate ions causes an electrostatic effect.
6.1 INTRODUCTION

The purpose of this chapter is to report an experimental comparison of the main properties of water transport in fired clay brick: capillary suction and water absorption porosities. These properties are of great practical importance because in masonry these are the main parameters associated with the presence and movement of water and hence on the porous material’s ability to dewater wet mixes.

This chapter begins with a review on the importance of water movement in clay bricks and its great effects on their performance. Then experimental work is presented comparing sorptivity with the initial rate of absorption (1 minute test), and then comparing water absorption measured at different soaking times with the vacuum saturation porosity and satiation porosity for three types of brick.

The time for water to travel to the centre of the brick and the proportion of saturation achieved using the results from the water absorption measurements were calculated and presented. This has implications for the design of the “brick mould” used in dewatering mortars.

6.2 BACKGROUND

Water movement in porous building materials like clay bricks has a large influence on their performance, and is the main cause of many practical problems such as rising damp, frost damage, efflorescence, and sulphate attack in mortar. The total porosity of clay bricks has a major effect on compressive strength, and permeability. Thus, in the specification of clay bricks, water absorption is always taken as a guide for the prediction of durability. The capillary suction properties of clay bricks also have an effect on both construction practice and weathering performance. Water suction is responsible for the bonding between mortar and brick. The higher the suction, the more rapid the removal of water from the fresh mortar and this can cause incomplete hydration of hydraulic binder and result in a weak or porous mortar. Low suction, typical of engineering bricks, will result in quick surface saturation and the excess water
reduces the bonding capacity of the mortar to the brick and then durability may be reduced [46].

The capillary suction properties also explain the behavior in driving rain. Bricks having high suction absorb rain water quickly and their surface saturation time is relatively long. Up to the point of surface saturation, all falling rain is absorbed and no run-off occurs. Low sorptivity bricks on the other hand have a short time to surface saturation meaning that overflow will start to occur relatively quickly leading to water penetration through perpendicular joints or defects [120, 121].

For these reasons it is necessary to know the water absorption characteristics or porosity, as well as the capillary suction properties of masonry materials. It is well established [28-31] that sorptivity is the most useful parameter to describe the capillary water absorption properties of a porous solid.

**6.3 EXPERIMENTAL DETAILS**

**6.3.1 Clay brick materials**

Three types of fired clay bricks supplied by Ibstock Brick Limited were used in this study (detailed description is given in chapter 3). Briefly two types were machine-made pressed facing bricks, (Leicester Buff and Golden Purple). The other (Staffordshire Blue) was a wire-cut extruded engineering brick. The extruded Staffordshire Blue brick is dark blue-grey in colour and had no perforations. The pressed bricks are buff in colour and had a single frog. Figure 6.1 shows a brick of each type. The same sets of bricks (10 bricks in all) were used in all measurements.

![Figure 6.1: The three types of brick used in the experimental work: left, Golden Purple; middle, Leicester Buff and right, Staffordshire Blue.](image)
6.3.2 Water absorption test methods

Two methods designed to measure the capillary water absorption properties of brick were compared: the sorptivity test and the 1 minute initial rate of water absorption test according to BS EN 772-11:2011 [96] (detailed descriptions of both are in chapter 3). Sorptivity was measured on each of the three faces (bed, stretcher and header face) on 3 bricks of Leicester Buff and Golden Purple and on 4 bricks of Staffordshire Blue. The sorptivity $S$ (mm min$^{-1/2}$) of an initially dry sample was determined from the gradient of a graph of the volume of water absorbed per unit area $i$ (mm) versus the square root of time $t^{1/2}$ (min)$^{1/2}$.

The experimental arrangement recommended for the determination of the 1 min initial rate of water absorption of clay masonry units is similar to that for sorptivity. Thus the initial rate of absorption values reported here were not measured in a separate set of experiments but were calculated from the masses at 1 minute obtained during the sorptivity measurements.

For porosity measurements, three methods were used: the 24 hours soak water absorption test according to BS EN 772-21:2011 [97]; vacuum saturation porosity; and satiation porosity (detailed descriptions of the three methods are given in chapter 3). In brief, the water absorption according to BS EN 772-21:2011 is defined as the percentage by mass of pore volume filled in a 24 h soak. In the experimental work reported here the percentage was also calculated for soaking times of 1 min, 9 min, 25 min, 1 h, and 72 h. For Staffordshire Blue brick the percentage was calculated also at 45 h and 2 weeks. Vacuum saturation porosity was used to assess the available volume of pore space of the open porosity of the three types of brick. In this method water is forced under pressure into pores and all the interconnected pore space becomes filled with water. The vacuum saturation porosity $f$ is calculated from the following equation:

$$f = \frac{\text{volume of water absorbed}}{\text{volume of sample}} \times 100\%$$  \hspace{1cm} (6.1)

As the pressed bricks have a frog the volume of the pressed bricks was measured in two ways: one by measuring the dimensions then calculating the volume of the frog and then subtracting this from the total volume of brick; the other by measuring the volume of the brick as an irregular shaped object by immersing it in an “Archimedes displacement vessel” filled with water. After the brick was submerged the amount of water displaced by the brick was weighed. The volume of this water was equal to the volume of the brick.
Satiation porosity is the volume fraction of pores occupied by water after absorption through one face (as in the determination of sorptivity) has reached the top of the sample. The satiation porosity determined from one-dimensional absorption is less than the vacuum saturation porosity where all the air in available pore space has been replaced by water. In practice, water absorption results in incomplete filling of pores and some pores remaining containing air. In this work satiation porosity is denoted by $f'$ and it is also calculated using equation (6.1).

6.4 RESULTS AND DISCUSSION
6.4.1 Capillary suction properties
6.4.1.1 Sorptivity results
The water absorption data (plotted as $i$ versus $t^{1/2}$) measured through each face of the three types of brick examined is shown in figures 6.2. For pressed bricks three bricks were measured on each face and for the extruded bricks four bricks were measured on each face. Each plot shows clearly that the mass of water absorbed per unit area increases linearly with the square root of time. The linearity of the relationship for all samples was checked by the value of the coefficient of linear correlation $R^2$ which was greater than 0.99. A relation is considered to be sufficiently linear if the value of $R^2 > 0.96$ [122]. It can be seen from the figure that there is little variation between samples for the Leicester Buff and Golden Purple bricks or between the stretcher and bed faces for these bricks. For the extruded brick, the sorptivity is very much lower as shown by the scale of the $y$ axis.

Sorptivity, which is the gradient of the graph of $i$ versus the square root of time $t^{1/2}$, measured through each face (bed, stretcher and header) for the three types of brick examined are summarised in table 6.1. The table also shows the mean and standard deviation. The low standard deviations show that sorptivity is a reproducible measure for masonry brick units.
Figure 6.2: Variation in $i$ versus $t^{1/2}$ at each face for three samples of Leicester Buff and Golden Purple bricks and four samples of Staffordshire brick. $i_1$, $i_2$, $i_3$, and $i_4$ refer to samples 1, 2, 3, and 4 respectively.
Table 6.1: Sorptivity values of the three faces of each brick type.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sorptivity (mm min$^{-1/2}$)</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Bed face</td>
<td>Stretcher face</td>
<td>Header face</td>
<td></td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>2.69</td>
<td>2.60</td>
<td>2.76</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>2.53</td>
<td>2.65</td>
<td>3.00</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>2.59</td>
<td>2.51</td>
<td>2.33</td>
</tr>
<tr>
<td>Mean</td>
<td>2.60</td>
<td>2.59</td>
<td>2.70</td>
<td></td>
</tr>
<tr>
<td>Standard Deviation</td>
<td>0.08</td>
<td>0.07</td>
<td>0.34</td>
<td></td>
</tr>
</tbody>
</table>

Leicester Buff

Golden Purple

|            | 1                           | 2.93   | 2.82   | 2.82   |
|            | 2                           | 2.75   | 2.60   | 3.12   |
|            | 3                           | 2.96   | 2.64   | 3.01   |
| Mean       | 2.88                        | 2.69   | 2.98   |
| Standard Deviation | 0.11               | 0.12   | 0.15   |

Staffordshire Blue

|            | 1                           | 0.07   | 0.02   | 0.01   |
|            | 2                           | 0.07   | 0.01   | 0.01   |
|            | 3                           | 0.07   | 0.01   | 0.01   |
|            | 4                           | 0.06   | 0.01   | 0.01   |
| Mean       | 0.07                        | 0.01   | 0.01   |
| Standard Deviation | 0.005           | 0.005   | 0     |

Figure 6.3 presents the variation in $i$ versus $t^{1/2}$ of the three faces of each type of brick in one plot, where each point is the mean value of three samples. The sorptivity of the header face is slightly higher than that of the other two faces for both pressed bricks, but in the extruded type sorptivity is much higher in the bed face than in the header and stretcher faces. This is no doubt associated with variation produced during the manufacturing processes. There is less dense material at the ends in the pressed bricks which explains the slightly higher sorptivity of the header face, while in the extruded brick less dense material is more probable along the cut surface which is the bed face. These different values of sorptivity in different directions reveal a clear anisotropy in clay bricks, some types showing this anisotropy more than others.
Figure 6.3: Variation in sorptivity of the three faces of brick (mean value of three samples). $i_h$, $i_s$, and $i_b$ refer to bed, stretcher, and header faces respectively.

### 6.4.1.2 Initial rate of absorption

Figure 6.4 shows the variation of $i$ versus $t^{1/2}$ of the bed face (mean value of three) of each type of brick using two $y$ axes at different scales. The left hand axis refers to the 1 minute initial rate of absorption which is the gradient of the line between $t^{1/2} = 1$ min$^{1/2}$ to the origin. The right hand axis refers to the bed face sorptivity which is the gradient
of the line between $t^{1/2} = 1$ to $t^{1/2} = 5$ minutes. The value of $i$ at $t^{1/2} = 1$ is the same for the two lines but drawn in different scales.

![Graphs of Leicester Buff, Golden Purple, and Staffordshire Blue Bricks](image)

Figure 6.4: Variation in $i$ versus $t^{1/2}$ of the bed face (mean value of three bricks) for the three types of brick. The linear line equations are shown for each set of data: ● initial rate of absorption; ■ bed face sorptivity.

Figure 6.4 shows clearly that the 1 min initial rate of absorption values for all three brick types is higher than the corresponding sorptivity which may be due to the positive intercept in all $i$ versus $t^{1/2}$ graphs although they show the same trend of sorptivity in that the initial rate of absorption of the pressed bricks is much higher than that of the extruded type. The initial rate of absorption ($I$) is numerically equal to the sorptivity, $S$, if the straight line graph of $i$ versus $t^{1/2}$ passes through the origin but this rarely happens. Gummerson et al. [123] showed that the sorptivity of most bricks produced a positive
intercept. Initial rate of absorption values increase significantly with immersion depth of sample because the latter allows some water absorption through the sides of the sample as well as the base [8].

Table 6.2 summarises the values of the initial rate of water absorption (1 min). The bed face sorptivity of the three brick types is repeated from table 6.1 for comparison. The mean and standard deviation values are recorded in each case.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Initial rate of absorption (kg m(^{-2})min(^{-1}))</th>
<th>Sorptivity (mm min(^{-1/2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>Leicester Buff</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>3.30</td>
<td>2.69</td>
</tr>
<tr>
<td>2</td>
<td>3.18</td>
<td>2.53</td>
</tr>
<tr>
<td>3</td>
<td>2.90</td>
<td>2.59</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>3.13</td>
<td>2.60</td>
</tr>
<tr>
<td><strong>Standard Deviation</strong></td>
<td>0.21</td>
<td>0.08</td>
</tr>
<tr>
<td></td>
<td><strong>Golden Purple</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>3.33</td>
<td>2.93</td>
</tr>
<tr>
<td>2</td>
<td>4.13</td>
<td>2.75</td>
</tr>
<tr>
<td>3</td>
<td>2.97</td>
<td>2.96</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>3.48</td>
<td>2.88</td>
</tr>
<tr>
<td><strong>Standard Deviation</strong></td>
<td>0.59</td>
<td>0.11</td>
</tr>
<tr>
<td></td>
<td><strong>Stafford Shire Blue</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>0.17</td>
<td>0.07</td>
</tr>
<tr>
<td>2</td>
<td>0.16</td>
<td>0.07</td>
</tr>
<tr>
<td>3</td>
<td>0.16</td>
<td>0.07</td>
</tr>
<tr>
<td>4</td>
<td>0.17</td>
<td>0.06</td>
</tr>
<tr>
<td><strong>Mean</strong></td>
<td>0.17</td>
<td>0.07</td>
</tr>
<tr>
<td><strong>Standard Deviation</strong></td>
<td>0.006</td>
<td>0.005</td>
</tr>
</tbody>
</table>

There is little variation between samples for Leicester Buff brick. For Golden Purple the variation is higher, while for the extruded brick there is very little or almost no variation between individual samples. The value of the initial rate of absorption is consistently higher than sorptivity and overestimates the true suction of the bricks. It is clear that 1 minute is not enough time to describe the brick capillary suction and its real interaction with the adjacent mortar.

From above it can be seen that the BS EN 772-11:2011 standard method for the 1 minute capillary absorption test which uses one data point only can give misleading results. The sorptivity is seen as a more accurate measurement of capillary water absorption ability although it takes longer time to determine. However the 1 min test
could be much improved by simply collecting data for several times and plotting these versus time$^{1/2}$.

6.4.2 Water absorption porosities

6.4.2.1 24 h soaking water absorption

Although this test method for porosity measurement is the most widely used in the brick industry, it is based on a mass percentage rather than volume percentage. It gives a smaller number because the density of water is much less than that of brick.

The water absorption percentage by mass for different soaking times from 1 minute to many hours for the three types of bricks is shown in Table 6.3 and presented as a histogram in figure 6.5. The water absorption of the two pressed types is much higher at all times than that of the extruded brick.

Leicester Buff absorption in 24 hours is 19% which is similar to the Golden Purple absorption of 16%. After 1 minute soaking Leicester Buff absorption is 50% of the 24 hour absorption, and then increases to 88% of the 24 hour value in 25 minutes and 90% of this absorption is completed in 1 hour. Further increase in water absorption after 24 hours is very small. After 72 hours it increases by less than 4% of the 24 hour absorption.

Table 6.3: Water absorption values of the three types of bricks for different soaking times.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>water absorption % by mass</th>
<th>Soaking time</th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1 min</td>
<td>9 min</td>
<td>25 min</td>
<td>1 h</td>
<td>24 h</td>
<td>45 h</td>
<td>72 h</td>
</tr>
<tr>
<td><strong>Leicester Buff brick</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>9.65</td>
<td>15.91</td>
<td>17.09</td>
<td>17.25</td>
<td>19.11</td>
<td>19.89</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>9.48</td>
<td>15.40</td>
<td>16.73</td>
<td>17.03</td>
<td>19.43</td>
<td>20.11</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>9.60</td>
<td>15.65</td>
<td>17.15</td>
<td>17.22</td>
<td>19.13</td>
<td>19.85</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>9.58</td>
<td>15.65</td>
<td>16.99</td>
<td>17.17</td>
<td>19.23</td>
<td>19.95</td>
<td></td>
</tr>
<tr>
<td><strong>Golden Purple brick</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>9.18</td>
<td>15.37</td>
<td>15.49</td>
<td>15.52</td>
<td>15.99</td>
<td>16.43</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>9.17</td>
<td>15.82</td>
<td>16.23</td>
<td>16.40</td>
<td>16.87</td>
<td>17.29</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>8.00</td>
<td>14.53</td>
<td>14.78</td>
<td>14.83</td>
<td>15.34</td>
<td>15.77</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>8.78</td>
<td>15.24</td>
<td>15.50</td>
<td>15.58</td>
<td>16.07</td>
<td>16.50</td>
<td></td>
</tr>
<tr>
<td><strong>Staffordshire Blue brick</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td></td>
<td>0.20</td>
<td>0.29</td>
<td>0.41</td>
<td>0.53</td>
<td>2.05</td>
<td>2.15</td>
<td>2.76</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td>0.26</td>
<td>0.44</td>
<td>0.62</td>
<td>0.78</td>
<td>2.91</td>
<td>2.94</td>
<td>3.26</td>
</tr>
<tr>
<td>3</td>
<td></td>
<td>0.23</td>
<td>0.33</td>
<td>0.48</td>
<td>0.63</td>
<td>2.63</td>
<td>2.85</td>
<td>3.16</td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>0.23</td>
<td>0.35</td>
<td>0.50</td>
<td>0.65</td>
<td>2.53</td>
<td>2.65</td>
<td>3.06</td>
</tr>
</tbody>
</table>
The water absorption of Golden Purple brick is lower than that of Leicester Buff and shows a similar pattern with soaking time. 1 minute absorption is 55% of 24 hours absorption, then 95% of the 24 hours absorption is completed in 9 minutes and reaches 97% in 25 minutes. Also further absorption after 24 hours is small: after 72 hours it increases by less than 3% of the 24 hour value.

For the extruded brick, water absorption is very much lower than for the pressed types. The 24 hours absorption, $w_m$, is 2.5% compared with 19% and 16%. Absorption is also very slow during the first hour. The 1 minute absorption is less than 10% of 24 hour absorption; increases to 20% in 25 minutes and reaches 25% of the 24 hour value after 1 hour. However absorption continues with time and increases by more than 20% of the 24 hour value after 72 hours and reaches 30% more after two weeks.

These results show clearly that time for full water absorption by immersion is different for different brick types and that a fixed time for this test is not appropriate.

It is worth saying that the 24 h water absorption values of all the types of bricks are within the limits given by the manufacturer (Table 3.1 of chapter 3) which quotes absorption $\leq 25\%$ for Leicester Buff, $\leq 20\%$ for Golden Purple, and $\leq 7\%$ for Staffordshire Blue.
6.4.2.2 Vacuum saturation porosity

The data of the vacuum saturation porosity measurements provide a true porosity, i.e. percent by volume, unlike the previous section. Although care has been taken not to call the (24 hour soak) a porosity, many others do refer to it this way or use it as substitute for porosity. Table 6.4 shows the vacuum saturated porosity of the three types of brick after 30 minutes vacuum followed by 24 hour immersion.

The porosity for the pressed types is high (46% Leicester buff, and 44% Golden Purple), and much higher than the porosity of the extruded brick (11%). The porosity of Leicester Buff bricks is higher than that of Golden Purple bricks, although the sorptivity of the latter is higher meaning that porosity alone does not explain water movement within brick.

Table 6.4: Vacuum saturation porosity of the three types of brick.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Vacuum saturation porosity %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Leicester Buff brick</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>45.52</td>
</tr>
<tr>
<td>2</td>
<td>45.96</td>
</tr>
<tr>
<td>3</td>
<td>46.10</td>
</tr>
<tr>
<td>Mean</td>
<td>45.86</td>
</tr>
<tr>
<td><strong>Golden Purple brick</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>43.76</td>
</tr>
<tr>
<td>2</td>
<td>43.74</td>
</tr>
<tr>
<td>3</td>
<td>44.66</td>
</tr>
<tr>
<td>Mean</td>
<td>44.05</td>
</tr>
<tr>
<td><strong>Staffordshire Blue brick</strong></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>10.65</td>
</tr>
<tr>
<td>2</td>
<td>10.95</td>
</tr>
<tr>
<td>3</td>
<td>10.93</td>
</tr>
<tr>
<td>Mean</td>
<td>10.87</td>
</tr>
</tbody>
</table>

The amount of water absorbed by the bricks during this method is much higher than the absorption by soaking test. The 24 hour soak water absorption porosity represents only 42%, 37% and 23% of vacuum saturation porosity for the Leicester Buff, Golden Purple and Staffordshire Blue bricks respectively. This is due to 24 h being insufficient.
6.4.2.3 Satiation porosity (effective porosity)

Table 6.5 shows the satiated porosity of the three types of brick. The satiated porosity of the bricks was 29% for Leicester Buff, 25% for Golden Purple and 3% for the Staffordshire engineering brick.

<table>
<thead>
<tr>
<th>Brick Type</th>
<th>Satiation porosity %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leicester Buff</td>
<td>29</td>
</tr>
<tr>
<td>Golden Purple</td>
<td>25</td>
</tr>
<tr>
<td>Staffordshire Blue</td>
<td>3</td>
</tr>
</tbody>
</table>

These values are much less than the values of the vacuum saturation porosity, which gives proof that not all the interconnected pore space becomes filled with water and some pores remaining contain air.

The phenomenon of rising damp in clay bricks is a result of long-term capillary rise. An earlier study [124] found that the arrival of water to the top of a brick by capillary rise does not correspond to the saturated state, and it takes 2 years for common clay brick to absorb the same amount of water as would be achieved by vacuum saturation porosity measurement.

6.4.3 Time needed for water to reach the brick centre

The time needed for water to travel to the centre of a brick was calculated because soaking time greater than this will add no further water. These measurements also give a guide to how long the soaking time should be.

The sorptivity as stated before is determined using equation 6.2 (equation 2.18 of chapter 2) below:

\[ i = S t^{1/2} \quad (6.2) \]

The distance \( x \) travelled by the wet front in the brick is given in Hall and Hoff, 2002 [7] as

\[ x = \frac{i}{f} \quad (6.3) \]

So that

\[ x = \frac{s}{f} t^{1/2}. \quad (6.4) \]
Squaring each side of equation 6.4 and rearranging gives

\[ t = \left( \frac{f' x}{s} \right)^2 \]  \hspace{1cm} (6.5)

where \( t \) = Time for the wet front to travel a distance \( x \),
\( f' = \) Satiation Porosity

The shortest distance to centre of a brick in each case was approximately 33 mm (half the header depth of a brick). Using this value for \( x \) and the values for \( S, f \) and \( f' \) (both vacuum saturation porosity and satiation porosity) for each brick type from Tables 6.2, 6.4 and 6.5 allows calculation of the time taken for the wet front to reach the centre of the brick in each case. These values are summarised in table 6.6.

Table 6.6: Travelling time of the wet front to the centre of the three types of brick.

<table>
<thead>
<tr>
<th>Brick Type</th>
<th>Time for wet front to reach centre of brick based on vacuum saturation porosity (min)</th>
<th>Time for wet front to reach centre of brick based on satiation porosity (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Leicester Buff</td>
<td>34</td>
<td>14</td>
</tr>
<tr>
<td>Golden Purple</td>
<td>26</td>
<td>8</td>
</tr>
<tr>
<td>Staffordshire Blue</td>
<td>2700 (45 hr)</td>
<td>200 (3.5 hr)</td>
</tr>
</tbody>
</table>

The time for the wet front to reach the centre of the brick was longer if the vacuum saturation porosity value was used instead of the satiation porosity.

These values are supported by the results obtained from water absorption at different soaking times. The water absorption for Leicester Buff brick gives 88% of the 24 hours absorption in 25 minutes and the calculation gives 34 minutes for full absorption. For Golden Purple water absorption results gives 97% of the 24 hour absorption in 25 minutes and the calculation gives 26 minutes.

It is also clear for the extruded brick that the 24 hour water absorption is not enough time for full absorption, and the calculation gives 45 hour (nearly 2 days) for full absorption.

From the results shown in table 6.6, it is found that the time for full water absorption is different for different brick types: while the pressed brick needs less than one hour for full absorption the extruded brick needs 2 days. Therefore the water absorption standard EN BS 772-21:2011 needs to be revised to take account of this.
6.4.4 Proportion of saturation at 24 hours soaking

The proportion of saturation for the three types of brick after 24 hours immersion was calculated from the mass of water absorbed in 24 hours divided by the vacuum saturation mass of water for that brick. This gave 69% saturation for Leicester Buff, 73% for Golden Purple, and 55% for Staffordshire Blue.

These proportions indicate that the amount of water absorbed in 24 hours fills about 70% of the pores of the pressed brick and fills slightly more than half the pores of the extruded brick. The remaining pore space must still be occupied by air, which at longer times will diffuse out.

These results give, for the first time, an estimate of how the water absorption test of pressed bricks compares with the saturated porosity, i.e. how a set of mass data can be translated into volume and become estimates of porosity.

For the extruded brick the 45 hour water absorption is measured for three samples and the mean value is 2.65%, hence at this time about 60% of the pores are filled with water and so air fills about 40% of the available pores.

Table 6.7 summarises the water absorption parameters determined for the three types of brick used in this work.

Table 6.7: Summary of the water absorption parameters obtained from the experimental work.

<table>
<thead>
<tr>
<th>Water absorption parameter</th>
<th>Leicester Buff</th>
<th>Golden Purple</th>
<th>Staffordshire Blue</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sorptivity (mm min(^{-1/2}))</td>
<td>2.6</td>
<td>2.88</td>
<td>0.07</td>
</tr>
<tr>
<td>Initial rate of absorption (mm min(^{-1}))</td>
<td>3.13</td>
<td>3.48</td>
<td>0.16</td>
</tr>
<tr>
<td>24 hr water absorption % by mass</td>
<td>19</td>
<td>16</td>
<td>3</td>
</tr>
<tr>
<td>Vacuum saturation porosity % by volume</td>
<td>46</td>
<td>44</td>
<td>11</td>
</tr>
<tr>
<td>Satiated porosity % by volume</td>
<td>29</td>
<td>25</td>
<td>3</td>
</tr>
<tr>
<td>Proportion of saturation at 24 hr soak %</td>
<td>69</td>
<td>73</td>
<td>55</td>
</tr>
<tr>
<td>Wet front travelling time based on vacuum saturation porosity (min)</td>
<td>34</td>
<td>26</td>
<td>2700</td>
</tr>
<tr>
<td>Wet front travelling time based on satiation porosity (min)</td>
<td>14</td>
<td>8</td>
<td>200</td>
</tr>
</tbody>
</table>
6.5 CONCLUSIONS

- Sorptivity may vary through the different faces of brick, *i.e.* bricks show anisotropy to water absorption and transmission. The sorptivity of pressed bricks is in general much higher than for extruded engineering brick. For both pressed bricks the sorptivity of the header is higher than that of the other two faces, but in the extruded type sorptivity is much higher in the bed face than in the header and stretcher faces. There is little variation between samples for the same pressed bricks. For extruded engineering brick, although the sorptivity is very much lower, there can be more variation between individual samples.

- The initial rate of absorption (1 min) test gives higher values than the sorptivity when expressed in the same units and this can be understood when the values of $i$ versus $t^{1/2}$ graphs give a positive intercept. It has shown that the 1 minute absorption test can give misleading results and sorptivity is seen as a more accurate indication of water absorption ability although it takes longer time to determine. However the 1 min test could be much improved by simply collecting several data points and plotting these versus time$^{1/2}$.

- Water absorption of pressed types is much higher at all times of soaking than that of extruded engineering brick. Time for full water absorption by immersion is different for different brick types; while pressed brick needs less than one hour for full absorption extruded brick needs nearly 2 days. Therefore the immersion time needs to be selected in the test procedures and standards, and the water absorption standard BS EN 772-21:2011 needs to be revised and improved.

- The satiated porosity is generally much less than the vacuum saturation porosity. The proportion of saturation indicates that the amount of water absorbed in 24 hour fills about 2/3 of the pores of pressed brick and fills slightly more than half the pores of extruded brick.

- It is found also that porosity alone does not predict or explain water movement within brick. For example the porosity of Leicester Buff bricks is higher than that of Golden Purple bricks, although the sorptivity of the latter is higher.
CHAPTER 7  EFFECT OF DEWATERING ON THE PROPERTIES OF FRESH AND HARDENED HYDRAULIC MORTARS

7.1 INTRODUCTION

Previous chapters (chapters 5 and 6) have considered the water transport properties of mortar and substrate (brick) individually. This chapter now considers the movement of water between the two in combination as in construction. It is an investigation of the inter-relationship between absorbent substrate (brick) and the water retaining ability of freshly mixed lime and cement mortars.

The experimental work of this study reports an investigation of the effect of dewatering, by the capillary suction of common clay facing bricks, on the setting time, sorptivity and strength of mortars prepared from natural hydraulic lime NHL5 and Portland cement CEM I. There is no published research neither on the effect of dewatering on the setting time nor on the sorptivity of cement or lime mortars, thus this is the first study to examine the effect of dewatering on these important fresh and hardened properties of hydraulic mortars in both the freshly-mixed and hardened states.

In order to examine the properties of hydraulic mortars as in practice after being dewatered by a substrate, an absorbent brick mould was designed to produce mortar bars of dimensions 40x40x160 mm which could be dewatered prior to setting. The time to dewater (\(t_{dw}\)) a fixed depth of mortar and the depth of brick needed were calculated using data from previous research. The same mixes were also cast in steel moulds which did not allow dewatering. The setting time of both dewatered and non-dewatered cement and lime mortars was measured. Compressive and flexural strength and sorptivity of hardened mortars from the same mix cast in both steel (non-dewatering) and brick (dewatering) moulds were measured at age of 7, 28, 56, 90 and 180 days.

7.2 BACKGROUND

The water desorption of fresh mortar by the substrate depends on both sorptivity of the substrate and desorptivity of the fresh mix [5]. The absorption of the substrate and the water retentivity of the wet mix are in competition and have significant influence on the fresh and hardened mortar properties such as workability, setting time, adhesion, sorptivity, strength and as a result on mortar durability.
In practice water is abstracted from the wet mix by the substrate (brick). Therefore mortars that are cast in steel moulds for the purpose of testing are not representative of the same mortars in their as-used states.

It was found with the API pressure cell that the gas breakthrough occurred before full dewatering of the wet mix had taken place. This means that, whilst the pressure cell could be used efficiently to measure desorptivity, it could not be used to prepare fully dewatered mortar samples for testing such properties as setting time or strength. Fully dewatered samples thus had to be prepared by dewatering the wet mixes by means of placing them in contact with a sufficient depth of absorbent substrate material.

The effect of dewatering on the compressive strength of cement and lime mortars has been studied and significant differences have been found in mortar strength following dewatering in the fresh state [62, 69]. However, there is no published research on the effect of dewatering on setting time, flexural strength or the sorptivity of set cement or lime mortars. Thus this study is the first study to examine the effect of dewatering on these properties.

7.3 CALCULATIONS FOR MOULD DESIGN

Calculations were carried out to determine both the thickness of brick which is enough for full dewatering and the time required to dewater a fixed depth of wet mortar mix. A theoretical model [32] which defines the time to dewater a freshly mixed mortar was used (detailed description in section 2.2.7.4 of chapter 2).

Dewatering of 40 mm of mortar of 1: 2: 0.78 (binder: sand: water) mix proportions and sand grain size of (150 μm- 300μm) is considered. Golden Purple bricks were used because of their high sorptivity (chapter 6). The brick capillary pressure of 0.044 MPa (unpublished data), satiated (effective) porosity 25%, and sorptivity 2.88 mm/min\(^{1/2}\) (from Chapter 6) were used.

Dewatering of mortars by brick depends on the sorptivity of the brick \(S\) and desorptivity of the mortar \(R\). The desorptivity is different for cement and lime mortar of the same mix proportions, therefore the time to dewater and the thickness of brick are different although only one type of brick was used.
7.3.1 Calculations for CEM I binder

The time to dewatering, \( t_{dw} \), can be calculated from equations (7.1) and (7.2) which are (2.33) and (2.38) of chapter 2:

\[
\frac{1}{A^2} = \frac{1}{R^2} + \frac{1}{S^2} \tag{7.1}
\]

and

\[
t_{dw} = \left( \frac{L \alpha}{A} \right)^2 \tag{7.2}
\]

The desorptivity \( R \) of CEM I mortar at 0.044 MPa = 2.54 mm/min\(^{1/2} \) [32]. The sorptivity \( S \) for golden purple brick = 2.88 mm/min\(^{1/2} \) (chapter 6). From these the transfer sorptivity \( A \) can be calculated as follows:

\[
\frac{1}{S^2} = 0.12 \text{ and } \frac{1}{R^2} = 0.16. \text{ Substituting these values in equation (7.1) gives }
\]

\[
\frac{1}{A^2} = 0.28, \text{ and therefore } A = 1.89 \text{ mm/min}^{1/2}
\]

Equation (7.2) shows that \( t_{dw} \) increases proportionally with the thickness of mortar bed, \( L \), and with the parameter \( \alpha \) which is a variable describing the water: solids volume fractions in the fresh and dewatered states of the mortar. The time to dewater also depends on the transfer sorptivity, \( A \), and is inversely proportional to it. The calculated value of \( \alpha \) for CEM I is 0.21[32].

If a 40 mm wide sample of wet mortar is placed between two absorbent bricks, each brick will dewater a depth of 20 mm, so \( L = 20 \) mm. Substituting for \( L, \alpha \) and \( A \) in equation (7.2) gives \( t_{dw} = 4.95 \approx 5 \) minutes. This is the time required to dewater 20 mm of CEM I mortar.

Sorptivity is determined using 7.3 (equation 2.39, chapter 2) below:

\[
i = S t^{1/2} \tag{7.3}
\]

The length of the wetted zone of the substrate can be expressed by Hall and Hoff [7]:

\[
x = \frac{i}{f'}
\]

where \( f' \) is the satiated porosity of the substrate.

Therefore

\[
x = \frac{S}{f'} t^{1/2} \tag{7.4}
\]

Substituting \( f' = 25\% \), \( S = 2.88 \text{ mm/min}^{1/2} \) and \( t_{dw} = 5 \) minutes into equation 7.4, the thickness of brick \( x \) required to dewater 20 mm of CEM I mortar is:

\[
x = \frac{2.88 \times \sqrt{5}}{0.25} \text{ giving } x = 25.76 \approx 26 \text{ mm}.
\]
7.3.2 Calculations for NHL5 binder

The same equations were used to calculate \( t_{dw} \) and thickness of brick required to dewater an NHL5 mortar. The desorptivity of NHL5 mortar, \( R \), at gas pressure 0.044 MPa is 1.44 mm/min\(^{1/2}\) [32].

Therefore \( \frac{1}{R^2} = 0.51 \) and \( \frac{1}{s^2} = 0.12 \), which gives \( \frac{1}{A^2} = 0.63 \) and \( A = 1.26 \) mm/min\(^{1/2}\). \( \alpha = 0.17 \) for NHL5 [32] and \( L = 20 \) mm. Substituting for \( L, \alpha, \) and \( A \) in equation 7.2 gives \( t_{dw} = 7.28 \approx 8 \) minutes as the time required to dewater 20 mm of NHL5 mortar.

The distance travelled by the wet from \( x \) can be calculated from equation (7.4).

Substituting \( f' = 25\% \), \( S = 2.88 \text{ mm/min}^{1/2} \) for Golden Purple brick and \( t_{dw} = 8 \) minutes in equation (7.4) gives \( x = 33 \) mm. This is the depth of brick needed to dewater 20 mm of this NHL5 mortar.

From the above calculations it was found that the time required to dewater 20 mm of the CEM I and NHL5 mortars is less than 10 minutes and the depth of brick needed is less than 40 mm. That means the standard depth of brick of 65 mm is sufficient to allow complete dewatering.

7.4 MOULD DESIGN

A brick mould was designed to produce mortar bars of the same dimensions as the standard steel mould (40x40x160) mm of BS EN 1015-11 [70] but which are fully dewatered prior to setting. Detailed explanation of the mould preparation is given in section 3.5 of chapter 3.

7.5 EXPERIMENTAL WORK

7.5.1 Materials and mix design

The binder materials used in the experimental work were Hanson Cement ordinary Portland cement CEM I and St Astier natural hydraulic lime NHL5. This lime was chosen because it is the most hydraulic of the NHLs and its setting is mainly by hydration reactions with carbonation reactions having less effect. The chemical composition as oxides and physical properties of the materials used are given in chapter 3.

A single source of mortar sand which conforms to BS EN 13139-02 sieved to a fraction of 300-600 \( \mu \text{m} \) was used in all mixes to ensure consistency.
Golden Purple brick was used to dewater mortar bars because it has a high sorptivity. A single mix design of 1: 2: 0.78 (binder: sand: water) by volume was examined in this study. These mix proportions are recommended to give suitable workability in masonry construction.

### 7.5.2 Preparation of test specimens

Mortar from the same mix was transferred both to the steel mould and to the brick mould according to BS EN 196-1:2005 [108]. To prevent adhesion of the mortar the steel moulds were cleaned and the internal faces of the assembled moulds were lubricated with a thin layer of mineral oil. For the brick moulds the sides of the bricks were covered with filter paper before filling. Each mould was filled with mortar in two approximately equal layers, each layer being vibrated on a vibrating table for 1 minute. The excess mortar in each case was skimmed off with a palette knife to make the mortar surface plane and level with the top of the mould. Then the moulds were covered with polyethylene sheet. For setting time measurements of dewatered mortars, the mortar was left in the brick mould for the time $t_{dw}$ calculated for that binder material before being placed in the Vicat mould.

### 7.5.3 Curing

Cement mortar specimens were left in the moulds for 24 hours then de-moulded and stored under water at room temperature until the test time. The lime mortar specimens were left in the mould for 2 days then de-moulded and placed in sealed polyethylene bags for another 5 days and then placed in the air until the test time. This curing procedure is not according to the standard BS EN 1015- part 11 [70] which recommends to store specimens in a humidity chamber at 20±5 °C and 65% relative humidity. The aim of this study was to compare the strength and sorptivity of the dewatered samples with the non-dewatered and the samples had to be cured in the same way. In addition curing the samples in air represents real site conditions, as carbonation will take place.

In total 48 prisms (40x40x160 mm) of CEM I and NHL5 mortars (24 prisms each) were prepared for compressive and flexural strength measurement. These prisms were tested at 7, 28, 56, and 90 days (3 samples dewatered and 3 non-dewatered at each age for both mortars). In addition 16 prisms of CEMI and NHL5 mortars were prepared for determination of sorptivity. One sample of dewatered and one of non-dewatered mortar
Chapter 7: Effect of dewatering on the properties of fresh and hardened hydraulic mortars

of both types were tested at ages of 28, 56, 90 and 180 days (plus the time required for drying to constant mass) as shown in the next section.

7.5.4 Determination of hardened mortar sorptivity

Sorptivity of hardened lime and cement mortar prisms was determined using the simple standard procedure which has been detailed in chapter 3. The sorptivity of mortars can only be measured when the mortar prisms are partially dry. Capillary absorption does not occur in saturated materials and drying in an oven at temperatures of 105°C can initiate microcracking in cement-based materials which will affect the sorptivity results. For this reason the sorptivity of hardened mortars will depend on the initial water content and consistency throughout the specimen under test. It is important therefore to ensure a constant drying procedure for all specimens [125].

The cement and lime mortar prisms were dried to constant mass at 50°C. This temperature was adopted to avoid any alteration to the capillary pore structure that would be caused by higher drying temperature. The time needed to attain constant mass of dewatered and non-dewatered lime and cement mortars varied between 5 and 14 days.

7.5.5 Determination of setting time

Initial and final setting times were determined using the Vicat method (detailed description in chapter 3). The initial setting time is defined as the time elapsed since the mixing water was added to the binder to when the needle penetration is 4 mm from the base of the mould. The final setting time is defined as the time when there is less than 0.5 mm penetration of a different needle. Setting time was determined for the dewatered and non-dewatered cement and lime mortars at two different ambient laboratory temperatures at different times of year (15°C and 25°C). For the dewatered mortars, Vicat measurements were not begun until the wet mix had been fully dewatered in the brick mould.

7.5.6 Determination of flexural and compressive strength

Flexural strength measurements were carried out according to BS EN 1015-11 (detailed in chapter 3). Flexural strength testing was carried out on six samples of each mortar mix: three dewatered samples (brick mould) and three non-dewatered (steel mould). First the dimensions (width and depth) of the prisms were measured by a vernier
calliper. The flexural strength of mortar was then determined by three-point loading of hardened prism specimens until failure and calculated from equation (7.5) (also 3.13):

$$R_f = \frac{1.5 F_f l}{b d^2}$$

(7.5)

Where $R_f$ is the flexural strength in MPa; $F_f$ is the maximum load applied to the middle of the prism at fracture, in N; and $l$ is the distance between the supports (100 mm); and $b$ and $d$ are the dimensions of the sides of the section of the prism in mm.

Following the flexural test, each bar produced two pieces which were then used for compressive strength measurement. The compressive strength was measured on one of these two pieces and then calculated from equation 7.6 (also 3.14):

$$R_c = \frac{F_c}{b d}$$

(7.6)

Where $R_c$ is the compressive strength in MPa, $F_c$ the maximum load at fracture in N, $b$ the width of the auxiliary plate (40 mm) and $d$ the depth of the prism in mm. Results of strength are presented to ±0.01 MPa.

### 7.6 RESULTS AND DISCUSSION

Figure 7.1 shows CEM I and NHL 5 mortars bars 10 minutes after casting in the brick mould.

![Cement mortar and Lime mortar](image)

Figure 7.1: Cement and lime mortars 10 minutes after casting in 40x40x160 mm brick moulds.

The dark area on each side of the mould shows the water abstracted from the mortar. 20 mm depth of cement mortars dewatered in approximate 5 minutes and NHL5 mortars in approximate 8 minutes (as predicted in the calculations.) The mould was constructed of sufficient thickness of brick so that the wet mortar could be, in theory, fully dewatered prior to setting. It can be seen that the brick absorbs a higher amount of water from cement mortar than lime mortar because of the higher desorptivity of the cement binder. Previous work has shown that 40-60% of the water mix is lost on dewatering to clay
Chapter 7: Effect of dewatering on the properties of fresh and hardened hydraulic mortars

facing brick (as shown in chapter 4). Clearly the dewatered mortar in use will not be the same material as that cast in a steel mould for testing purposes.

7.6.1 Effect of dewatering on setting time

Comparison of the results obtained from the Vicat test for initial and final setting time of dewatered and non-dewatered CEM I and NHL5 mortars at 15 and 25°C are presented in table 7.1 and plotted in figure 7.2.

Table 7.1: Initial and final setting times of the dewatered and non-dewatered CEM I and NHL5 mortars at 15 and 25°C.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>CEM I mortar</th>
<th>NHL5 mortar</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Non-dewatered</td>
<td>Dewatered</td>
</tr>
<tr>
<td>Initial setting time</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>3h: 55min</td>
<td>45 min</td>
</tr>
<tr>
<td>25</td>
<td>3h: 35 min</td>
<td>35 min</td>
</tr>
<tr>
<td>Final setting time</td>
<td></td>
<td></td>
</tr>
<tr>
<td>15</td>
<td>6h: 12 min</td>
<td>2h: 24 min</td>
</tr>
<tr>
<td>25</td>
<td>5h: 48 min</td>
<td>2h: 12 min</td>
</tr>
</tbody>
</table>

Figure 7.2: Initial and final setting time for dewatered and as-mixed (non-dewatered) CEM I and NHL5 mortars at 15 and 25°C.

The results show clearly that the initial and final setting times of lime mortars are longer than for cement mortars. Initial setting time is the time needed to change from fluid to rigid stage [36], and final setting time represents the end of workability and the
Chapter 7: Effect of dewatering on the properties of fresh and hardened hydraulic mortars

beginning of hardening. Setting of hydraulic lime is slower than cement mortar because the dominant calcium silicate present in hydraulic lime is dicalcium silicate (C$_2$S) and not tricalcium silicate (C$_3$S), formation of which requires a higher kiln temperature. Initial and final setting times are greatly reduced for both lime and cement mortar by dewatering. The initial setting time of dewatered cement mortar at 15°C was reduced from 3.92 h to 0.75 h which represents an 80% reduction. The final setting time was reduced by a similar amount: from 6.2 h to 2.4h and represents 61% reduction. The dewatering effect on setting time of lime mortar is nearly the same. The initial and final setting of NHL5 mortar were reduced by approximately 6 hours at 15°C which represents 77% and 61% reduction of the initial and final setting times respectively.

It is well known that setting time is affected by temperature. It reduces as the temperature rises [38]. The results in figure 7.2 show that the reduction in setting time of dewatered CEM I and NHL5 mortars is greater at 25°C than at 15°C.

Although the Vicat test is not a fundamental measure of setting time, gives a mechanical definition of setting and does not give an obvious link to hydration degree, because it tracks the ability of a rod of a fixed diameter to penetrate a certain consistency of a wet mix. The test is, however, entirely suitable for the comparative results carried out in this work. It is worth noting that in the development of new more sophisticated setting time test methods [39, 40, 126], the fundamental issue of dewatered mortars being different to non-dewatered mortars has still not been addressed.

### 7.6.2 Effect of dewatering on flexural and compressive strength

Table 7.2 shows the values of the maximum load applied, the dimensions $b$ and $d$ of the sides of the samples and the flexural and compressive strengths of the 6 samples of CEM I mortar (three dewatered and three non-dewatered) tested at 7, 28, 56 and 90 days. Mean and standard deviation of the values at each age are also given. The results of NHL5 mortar are presented in table 7.3 in a similar way.

The results for both CEM I and NHL5 mortars show that both the flexural and compressive strength increased with age of the mortar. For cement mortar the compressive strength $R_c$ was approximately 3-4 times the flexural strength $R_f$ at each age. The lime mortar results also showed that the flexural and compressive strength increased with age of the mortar and compressive strength was approximately 2-3 times the flexural strength at each age.
Table 7.2: the dimensions, maximum load and failure strength for dewatered and non-dewatered CEM I mortar, (left) under flexural load; (right) under compressive load.

<table>
<thead>
<tr>
<th>Age (7 days)</th>
<th>mould type</th>
<th>Sample No</th>
<th>$d$ (mm)</th>
<th>$b$ (mm)</th>
<th>$F_t$ (kN)</th>
<th>$R_c$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brick</td>
<td>1</td>
<td>38.45</td>
<td>41.60</td>
<td>3.03</td>
<td>7.39</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>40.90</td>
<td>40.83</td>
<td>2.45</td>
<td>7.58</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>41.60</td>
<td>40.73</td>
<td>3.46</td>
<td>7.37</td>
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</tr>
<tr>
<td>Mean</td>
<td></td>
<td>40.32</td>
<td>41.05</td>
<td>3.31</td>
<td>7.45</td>
<td></td>
</tr>
<tr>
<td>Std</td>
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<td>0.48</td>
<td>0.25</td>
<td>0.12</td>
<td></td>
</tr>
<tr>
<td>Steel</td>
<td>1</td>
<td>40.66</td>
<td>39.52</td>
<td>2.59</td>
<td>5.95</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>40.73</td>
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<td>2.47</td>
<td>5.63</td>
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</tr>
<tr>
<td></td>
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<td>40.14</td>
<td>39.23</td>
<td>2.54</td>
<td>6.02</td>
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<tr>
<td>Mean</td>
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<td>39.48</td>
<td>2.53</td>
<td>5.87</td>
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</tr>
<tr>
<td>Std</td>
<td></td>
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<td>0.24</td>
<td>0.26</td>
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<table>
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<tr>
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<th>mould type</th>
<th>Sample No</th>
<th>$d$ (mm)</th>
<th>$b$ (mm)</th>
<th>$F_t$ (kN)</th>
<th>$R_c$ (MPa)</th>
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<tr>
<td>Brick</td>
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<td>42.29</td>
<td>41.63</td>
<td>4.94</td>
<td>9.95</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>37.30</td>
<td>41.95</td>
<td>3.91</td>
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<tr>
<td></td>
<td>3</td>
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<th>$b$ (mm)</th>
<th>$F_t$ (kN)</th>
<th>$R_c$ (MPa)</th>
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<tbody>
<tr>
<td>Brick</td>
<td>1</td>
<td>40.54</td>
<td>40.91</td>
<td>4.40</td>
<td>9.83</td>
<td></td>
</tr>
<tr>
<td></td>
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<td>41.60</td>
<td>38.06</td>
<td>5.07</td>
<td>11.55</td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
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<td>41.60</td>
<td>5.36</td>
<td>11.22</td>
<td></td>
</tr>
<tr>
<td>Mean</td>
<td></td>
<td>41.21</td>
<td>40.19</td>
<td>4.94</td>
<td>10.86</td>
<td></td>
</tr>
<tr>
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<td>1.88</td>
<td>0.49</td>
<td>0.91</td>
<td></td>
</tr>
<tr>
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<td>40.65</td>
<td>4.32</td>
<td>9.79</td>
<td></td>
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<tr>
<td></td>
<td>2</td>
<td>41.00</td>
<td>40.75</td>
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<td></td>
<td>3</td>
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<td></td>
<td>40.59</td>
<td>40.48</td>
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<tr>
<td>Std</td>
<td></td>
<td>0.36</td>
<td>0.38</td>
<td>0.43</td>
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<table>
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<th>Sample No</th>
<th>$d$ (mm)</th>
<th>$b$ (mm)</th>
<th>$F_t$ (kN)</th>
<th>$R_c$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
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<td></td>
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<tr>
<td></td>
<td>3</td>
<td>40.76</td>
<td>41.91</td>
<td>5.73</td>
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<tr>
<td>Mean</td>
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<td>5.31</td>
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<td>Std</td>
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<td>0.36</td>
<td>0.66</td>
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<td>40.33</td>
<td>40.61</td>
<td>4.12</td>
<td>9.37</td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>40.23</td>
<td>40.17</td>
<td>4.00</td>
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<td></td>
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<td>40.01</td>
<td>41.20</td>
<td>4.33</td>
<td>9.84</td>
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<td>0.16</td>
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<td>0.16</td>
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</tr>
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Table 7.3: the dimensions, maximum load and failure strength for dewatered and non-dewatered NHL5 mortar, (left) under flexural load; (right) under compressive load.

<table>
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</tr>
<tr>
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<td>40.93</td>
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<td></td>
<td>3</td>
<td>40.80</td>
<td>39.63</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>40.88</td>
<td>39.24</td>
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<td>Std</td>
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<td>Mean</td>
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<tr>
<td>Brick</td>
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<td>40.44</td>
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<td></td>
<td>2</td>
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<td>40.70</td>
<td>39.57</td>
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<tr>
<td></td>
<td>Std</td>
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<td>0.99</td>
</tr>
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<td>40.26</td>
<td>40.50</td>
</tr>
<tr>
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<td>40.40</td>
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<tr>
<td></td>
<td>3</td>
<td>40.20</td>
<td>40.46</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>40.24</td>
<td>40.45</td>
</tr>
<tr>
<td></td>
<td>Std</td>
<td>0.03</td>
<td>0.05</td>
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<tbody>
<tr>
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<td>Sample No</td>
<td>$d$ (mm)</td>
<td>$b$ (mm)</td>
</tr>
<tr>
<td>Brick</td>
<td>1</td>
<td>40.42</td>
<td>40.85</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>40.92</td>
<td>41.52</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>42.43</td>
<td>41.11</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
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<tr>
<td></td>
<td>Std</td>
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<td></td>
<td>Std</td>
<td>0.08</td>
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<table>
<thead>
<tr>
<th>mould type</th>
<th>Age (90 days)</th>
<th>mould type</th>
<th>Age (90 days)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Sample No</td>
<td>$d$ (mm)</td>
<td>$b$ (mm)</td>
</tr>
<tr>
<td>Brick</td>
<td>1</td>
<td>40.05</td>
<td>41.39</td>
</tr>
<tr>
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<td>2</td>
<td>41.50</td>
<td>40.70</td>
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<tr>
<td></td>
<td>3</td>
<td>41.41</td>
<td>41.47</td>
</tr>
<tr>
<td></td>
<td>Mean</td>
<td>40.99</td>
<td>41.19</td>
</tr>
<tr>
<td></td>
<td>Std</td>
<td>0.81</td>
<td>0.42</td>
</tr>
<tr>
<td>Steel</td>
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<td>40.36</td>
<td>38.86</td>
</tr>
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<td>2</td>
<td>40.15</td>
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<td>Mean</td>
<td>40.24</td>
<td>39.44</td>
</tr>
<tr>
<td></td>
<td>Std</td>
<td>0.11</td>
<td>0.57</td>
</tr>
</tbody>
</table>
Table 7.4 summarises the average values (of three) flexural and compressive strength measurements of the dewatered and non-dewatered lime and cement mortars at 7, 28, 56 and 90 days. These values are also plotted in figure 7.3.

Table 7.4: Average values of flexural and compressive strength of dewatered and non-dewatered CEM1 and NHL5 mortars.

<table>
<thead>
<tr>
<th>Mould type</th>
<th>$R_f$ (average of 3 samples) (MPa)</th>
<th>$R_c$ (average of 3 samples) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Age (days)</td>
<td>Age (days)</td>
</tr>
<tr>
<td>CEM1 Mortar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brick mould</td>
<td>7.45 9.99 10.86</td>
<td>23.82 35.08 39.78 42.49</td>
</tr>
<tr>
<td>Steel mould</td>
<td>5.87 8.36 9.43 9.48</td>
<td>17.36 30.62 36.89 40.42</td>
</tr>
<tr>
<td>NHL5 Mortar</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brick mould</td>
<td>0.38 0.75 1.22 1.22</td>
<td>0.90 1.47 2.30 2.57</td>
</tr>
<tr>
<td>Steel mould</td>
<td>0.08 0.22 0.23 0.22</td>
<td>0.30 0.44 0.57 0.58</td>
</tr>
</tbody>
</table>

Figure 7.3: Flexural and compressive strengths of dewatered and non-dewatered mortars. CEM I mortars (a) & (b); and NHL5 mortars (c) & (d). (Note y axis scales).

It can be seen from figure 7.3 that dewatered samples show a higher strength at all ages than the non-dewatered samples of cement and lime mortars. In each case the strength is increased following dewatering in the wet-state. The highest increase in both
compressive and flexural strength of dewatered cement samples is at 7 days (about 40% and 30%) respectively (figure 7.5 (a) & (b)). For NHL5 the compressive strength of dewatered NHL5 samples at 28 days is more than 3 times that of non-dewatered samples and at 56 days is more than 4 times but shows little further increase thereafter as shown in figure 7.5 (c). Flexural strength of dewatered samples at all ages is 3-4 times the non-dewatered as shown in figure 7.5 (d).

The increase in strength on dewatering for cement mortar is less as a percentage than that of lime mortar. Therefore the properties of fresh lime mortar such as water retention and setting time are highly important compared to cement mortar. The water retentivity of Portland cement mortar is much less than that of natural hydraulic lime NHL5 mortar as shown earlier, therefore the loss of water from cement mortar on dewatering is higher than the loss from the equivalent NHL5 mortar. The strength of cement at early age (7 days) increased by this abstraction of mix water therefore the difference is higher for the cement mortars at early age. These strength results therefore show the influence and importance of water content within the mix.

Microstructure and spectroscopic analysis by El-Turki et al. [69] indicate that the dewatering process affects strength by influencing the growth of silicates rather than the growth of calcium carbonate; that is by influencing hydration reactions at early ages rather than carbonation at later age. They suggest that increase in strength comes from consolidation of particles and a growth of silica phases in the water located between sand grains.

7.6.3 Effect of dewatering on sorptivity of hardened mortars

The dewatered (brick mould) specimens dried faster than the non-dewatered (steel mould) specimens. The lime mortar specimens were dried in a shorter time (5-9) days as they were cured in air until test age whilst the cement specimens, which were cured under water, took 6-14 days to dry depending on the age.

Figure 7.4 shows typical plots of cumulative absorbed volume of water divided by the area of the absorbing face, $i$, versus the square root $t^{1/2}$ of time for dewatered and non-dewatered cement and lime mortars at age 28, 56, 90 and 180 days plus the drying time.
These plots indicate that there is little variation in water absorption at different ages of mortar in dewatered cement and lime mortars. For the non-dewatered, the variation with age is bigger. It can be seen that the water absorption per unit area of cement mortar is much less than that of lime mortar. The dewatered samples of both cement and lime mortars have much less water absorption per unit area than the corresponding non-dewatered as is obvious from the y-axis scales.

The sorptivity (gradient of \( i \) against \( t^{1/2} \)) values are summarised in table 7.5 and plotted in figure 7.5.

**Table 7.5: Sorptivity values (mm/min\(^{1/2}\)) of dewatered and non-dewatered CEM I and NHL5 mortars at 28, 56, 90 and 180 days age.**

<table>
<thead>
<tr>
<th>Age (day)</th>
<th>CEM I mortar</th>
<th>NHL5 mortar</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Non-dewatered</td>
<td>Dewatered</td>
</tr>
<tr>
<td>28</td>
<td>0.47</td>
<td>0.09</td>
</tr>
<tr>
<td>56</td>
<td>0.61</td>
<td>0.12</td>
</tr>
<tr>
<td>90</td>
<td>0.49</td>
<td>0.12</td>
</tr>
<tr>
<td>180</td>
<td>0.65</td>
<td>0.12</td>
</tr>
</tbody>
</table>
Figure 7.5 shows that the sorptivity of both hardened mortars are greatly decreased by dewatering in the fresh state. The sorptivity of non-dewatered CEM I mortar is less than 1 mm/min$^{1/2}$ at all ages measured and it is much less than the sorptivity of non-dewatered NHL5 mortars (about 5 mm/min$^{1/2}$) at all ages. This is explained by the higher desorptivity of CEM I allowing more water to be abstracted by the absorbent moulds.

The dramatic reduction in sorptivity of hardened cement and lime mortars by dewatering in the fresh state was the same percentage (about 80%). The same suggestion mentioned earlier of the reason for strength increase by dewatering is given for sorptivity decrease. It is possibly a result of the consolidation of particles and a growth of hydration compounds in water located between sand grains.
7.7 CONCLUSIONS

The main conclusions of this chapter are:

- It is possible to dewater mortars to the same extent that they will be dewatered in use by using a suitably designed and constructed absorbent brick mould.

- The initial and final setting times of both cement and hydraulic lime mortars are greatly reduced by dewatering. This is especially seen at higher temperature.

- Dewatering results in a significant increase in both flexural and compressive strength.

- The higher the desorptivity the more water releasing the mortar and therefore the greater the effect of dewatering on both setting time and strength.

- The sorptivity of both cement and hydraulic lime mortars in the set and hardened state is greatly reduced by dewatering in the freshly mixed state.

- The standard steel mould used in testing the setting time or strength of mortars gives values that underestimate the time to set and the strength of mortars in masonry. Therefore there are implications for standards: mould material should be changed to dewatering material like brick or any other materials that has an equivalent capillary pressure to those of bricks in masonry. Test procedure for setting time test should also be changed, Vicat measurements should not begun until the wet mix has been fully dewatered in a dewatering mould. Finally the time to testing should be considered. Currently lime mortars are tested at 28 days as for cement mortars, and clearly this time is not long enough and requires consideration.
CHAPTER 8 AN INVESTIGATION OF A FUNDAMENTAL EQUATION FOR WATER TRANSPORT

8.1 INTRODUCTION
All the work discussed so far in this thesis is underpinned by Sharp Front theory. Although the fundamental Sharp Front equation \( S = (2\kappa f \Psi)^{1/2} \) was proposed at least 10 years ago by Hall and Hoff [7], nobody has been verify it. The main objective of the work of this chapter is to validate this equation experimentally. The symbols have their usual meaning. This requires comprehensive measurements of sorptivity, permeability, porosity and capillary pressure of the same sample of a single porous material. The basics of unsaturated capillary flow of liquids in porous materials are briefly reviewed in the background of the chapter.

The hydraulic properties sorptivity, porosity, permeability and capillary pressure which are the four parameters of the equation were obtained experimentally and the results discussed. Measurements were carefully performed on the same brick types used in previous chapters. Sorptivity and porosity were measured by their conventional methods and by other developed techniques. Saturated permeability was measured by the constant head method. The capillary pressure of water rise in a dry brick was obtained by measuring the equilibrium pressure of the air compressed ahead of the advancing wetting front.

Knowledge of sorptivity, porosity and saturated permeability leads to the calculation of capillary pressure which is very hard to measure experimentally although simple in principle. However values of capillary pressure were measured and the closeness to calculated values taken as validation of the equation.

8.2 BACKGROUND
Traditional construction materials such as brick, stone and concrete are porous and permeable. The transport of water through permeable materials whose water content is non-uniform and less than saturation is the principal reason for many problems of building construction. For masonry water movement first starts at construction stage as mortar is dewatered by the brick substrate. During the life of a building water movement occurs as absorption and penetration by exposure to rain, ground water,
condensation and humid air or water released in response to the drying effects of the atmosphere. Rising damp is an instance of water movement which is a hard problem to investigate.

Usually water absorption by building materials is predicted by measurements of porosity, either by vacuum saturation or in the past by boiling in water for several hours. Water transport measurements have normally been limited to measurements of permeability on saturated specimens. It must be highlighted that water absorption porosity or saturated permeability does not define complete understanding of water movement in porous materials. Knowledge of the fundamental hydraulic parameters could be particularly useful in helping to put the requirement of good construction practice on a sound footing.

Water flow in porous building materials is driven mainly and often completely by capillary forces. This unsaturated flow highlights that this is linked to differences in moisture content. The basic law which describes the flow of water through unsaturated porous materials is the extended Darcy’s law explained in chapter 2. The Sharp Front model proposed by Hall and Hoff [7] gives an influential, simplified and estimated method to the analysis of unsaturated capillary flow of liquids in porous materials. In Sharp Front theory it is assumed that during capillary absorption the wetted region behind the wet front is uniform and has constant water content and the location of the advancing wet front is marked by a step change in liquid content. The Sharp Front model assumes that the capillary pressure at the wet front is constant and at the inflow face is zero. The application of this assumption (as shown in chapter 2) led to the derivation of equation 8.1 (which is also equation 2.37).

\[ S = (2KΨf)^{1/2} \]  

(8.1)

Where \( S \) is the sorptivity, \( K \) the saturated permeability, \( Ψ \) the wetting front capillary pressure and \( f \) the volume fraction porosity of the material. This is a fundamental and very powerful equation defining the advance of a wetting front into an initially dry porous solid by capillarity. Of the parameters in equation (8.1) \( Ψ \) is extremely difficult to measure whereas \( S, K \) and \( f \) are relatively easy to obtain. The purpose of the work presented here is to establish whether \( Ψ \) may be calculated from the more easily measured values of \( S, K \) and \( f \). Sorptivity is easy to measure by a simple procedure, described in full in chapter 3. The other two techniques for measuring sorptivity were also used in this study. These were the measurement of sorptivity by visual inspection.
of the advance of the wetting front and the measurement of sorptivity under vacuum (details in chapter 3).

Using the vacuum saturation porosity \( f \) in the equation assumes that the wetted zone is fully saturated and all the air present in the open porosity is expelled. In reality only a portion of the air existing in a dry material is displaced by capillary rise of the wetting front and the rest is trapped within the wetted region (unsaturated state). This state is represented by the satiation porosity \( f' \) whose numerical value is always less than \( f \). Saturated permeability can be determined directly according to Darcian flow. However in the real world construction materials are rarely completely saturated during construction or throughout their life time but it is very difficult to determine directly unsaturated hydraulic conductivity [7, 10].

Capillary pressure \( \Psi \) is very difficult to measure experimentally although it requires no complex equipment. Hall and Hoff [7] defined capillary pressure as the work performed against the capillary forces acting on the liquid. In the case of the absorption through one face of a sample with all other boundary surfaces unsealed, it is assumed that air driven forward by the advancing wetting front is displaced easily without generating a significant internal pressure gradient. The air pressure of the dry sample is considered uniform and equal to the atmospheric pressure. However in the case of absorption into a sample that is sealed on all faces except the inflow surface, the displaced air is driven ahead of the wetting front and trapped by the sealed end face and sides [7]. As the wetting front advances the resistance increases as it compresses the air in front of it. In the SF model the absorption stops when the pressure of the trapped air equals the wetting front capillary pressure. The wetting front capillary pressure of sealed clay bricks was obtained in this study by calculating the equilibrium pressure of the compressed trapped air in front of the advancing wetting front using an experimental technique based on the one described by Ioannou et al. [23].

Sorptivity and capillary pressure relate to the dry or partially saturated state because they will both be zero in the saturated state. Porosity and permeability can refer to the saturated or unsaturated state. The difficulty of the present study is to decide whether saturated or unsaturated conditions are relevant.
8.3 EXPERIMENTAL WORK

8.3.1 Materials
The same bricks used in previous chapters (Golden Purple and Leicester Buff bricks) were used. Two samples only from each type were used for all the experimental measurements of this chapter to eliminate the material variability. After each measurement it was necessary to dry each sample, therefore the experimental work took longer than expected. The same bricks were used at the beginning of the study as whole bricks (frogs only removed) for porosity and sorptivity measurements and then used as cut sections (cylinders and prisms) for permeability and capillary pressure measurements.

8.3.2 Porosity
Porosity was measured in the saturated and unsaturated state by three methods. Vacuum saturation porosity, satiation porosity and satiation porosity under vacuum were measured. The detailed description of the procedures of these methods is given in chapter 3. In a few words vacuum saturation porosity measures all the available volume of pore space of the open porosity and is denoted by $f$. In this method water is forced under pressure into pores and all the air in the interconnected pore space is replaced with water (saturated state). Satiation porosity (effective porosity), $f'$, measures the portion of pore space that is filled by capillary absorption (unsaturated state). Water is allowed to be absorbed from the bed face of the brick until it reaches the top using the same simple technique as sorptivity measurement but only being weighed once, when the water has reached the top. Vacuum satiation porosity, $f''$, is measured in the same way as $f'$ but with the initially dry sample inside a vacuum tank. The resistance of air pressure on the capillary rise of water is reduced in this way.

8.3.3 Sorptivity
The sorptivity of the bricks was measured by the standard sorptivity method, by visual sorptivity and by sorptivity under vacuum. A detailed description of the three methods is given in chapter 3. Briefly, sorptivity $S$ was first measured using the simple standard method described in Gummerson et al. 1980 [28]. The second method gave visual sorptivity $S_{vis}$ which was measured by visual inspection of the wet front, measuring the distance $x$ the water rose by capillarity with time instead of weighing. Two ways were
used to measure this distance (details in chapter 3). The third method was to measure sorptivity under vacuum $S_{vac}$. This method has been developed to eliminate the effect of air resistance on capillary rise. Each vacuum sorptivity measurement took at least one week because it requires the mass of the water absorbed to be obtained at a minimum of five time intervals (normally 1, 4, 9, 16, and 25 minutes). In vacuum sorptivity, after each time interval, the brick was weighed to 0.01 g and dried at 105 °C for at least 24 hours to constant mass.

### 8.3.4 Permeability

Saturated permeability was determined for two cylindrical specimens cut from each brick type parallel to bed and stretcher faces. The constant head method explained by Hall and Hoff [7] was used. The experimental arrangement produces simple Darcian flow through a liquid-saturated specimen. Briefly the method is to measure the mass of water to 0.01 g per unit area of the absorbing face (area calculated from measured diameter to 0.1 mm) per unit time. Unidirectional flow was assumed through the sample under the pressure of a known head of water. Cylindrical cores of fixed 20 mm diameter and depth between 21-24 mm were sealed with epoxy resin from the side surfaces and glued into the end of polythene tube of internal diameter 20 mm. This polythene tubing was used to provide the constant hydraulic head. The rate of flow $Q$ (mm$^3$/min) was determined for four heads of water (1000, 1250, 1500 and 1750 mm). The full description and schematic representation of the technique are given in chapter 3. The saturated permeability $K$ is defined by equation 8.2 [7] (also equation 3.10 in chapter 3).

$$K = \frac{QL}{H A} \quad (8.2)$$

Where $Q$ is the quantity of liquid per unit time flowing through the specimen of length $L$ and cross-sectional area $A$ under the action of constant hydraulic head $H$.

### 8.3.5 Capillary pressure

Capillary pressure of rectangular prisms cut from the same two bricks of each type with the long axis parallel to the bed and stretcher faces was found. Two prisms of dimensions 160*39*30 mm and 160*40*29 mm of Golden Purple brick and of dimensions 160*40*32 mm and 155*40*29 mm of Leicester Buff brick were used. After drying to constant mass in an air oven, surfaces were sealed with several layers of epoxy resin except the inflow surface. They were then fully immersed in water. The
sealed sides ensured that absorption occurred only perpendicular to the inflow face (detailed description and photographs are given in chapter 3).

In these initial experiments the increase in mass was measured by taking the sample out of the tank and weighing at regular intervals (daily) until there was no further change in the mass. The volume of water absorbed was determined from the mass increase, giving the volume of pores filled with water by capillary rise. Capillary pressure was determined using the ideal gas equation 8.3 (also 3.11) assuming the temperature is constant:

\[ p_1 v_1 = p_2 v_2. \]  

(8.3)

This was rearranged to equation 8.4 (also 3.12):

\[ p_2 = \frac{p_1 v_1}{v_2}. \]  

(8.4)

Here \( p_2 \) is equivalent to the pressure of the displaced air which is the pressure of the final volume of air (trapped air) \( v_2 \) left after capillary rise of water. \( p_1 \) is the pressure of the initial volume of air (pore space) \( v_1 \) in the brick and it is equal to the atmospheric pressure.

After these initial experiments a refinement was set up with a different weighing arrangement. The length of one prism of each type was shortened to 70 mm and was placed in an aluminium basket immersed in a water tank. The top of the basket was attached by a rigid copper wire to the weighing attachment at the base of an electronic Sartorius balance which was connected to a computer as shown in figure 8.1.

Figure 8.1: Schematic arrangement for measurement of capillary pressure.
Software (Sarto Connect) which enables the collection of data (mass of absorbed water) at constant time intervals was used. A time interval of 10 minutes was chosen. This refinement allowed the collection of early-time data which had not been possible in the preliminary method.

### 8.4 RESULTS AND DISCUSSION

The dimensions of sorptivity, $S$, are $L \ T^{-1/2}$ and of hydraulic conductivity, $K$, are $L \ T^{-1}$. Porosity $f$ has no dimensions. The capillary pressure $\Psi$ has dimension $L$ because the Sharp Front model assumes that the wetting front capillary pressure, $p_f$, is equivalent to the wetting front capillary potential as shown in equation 8.5

$$\Psi = \frac{-p_f}{\rho g}$$

(8.5)

where $\rho$ is the liquid density. The dimensions of $\Psi$ are shown below:

$$\Psi = \frac{p}{\rho g} = \frac{mg/L^2}{mg/L^3} = L.$$  

Thus the dimension of capillary pressure can be expressed as length. In this study the units of $S$ are mm/min$^{1/2}$, $K$ mm/min and $\Psi$ mm. using

$$S = (2Kf\Psi)^{1/2},$$

(8.1)

squaring each side of the equation gives

$$S^2 = 2KF\Psi.$$  

(8.6)

In terms of units

$$\frac{mm^2}{min} = 2 \frac{mm}{min} \times mm = \frac{mm^2}{min}.$$  

This analysis shows that equation 8.1 is dimensionally consistent.

#### 8.4.1 Porosity

The average porosity measured for duplicate samples by the three different methods for each type of brick are summarized in table 8.1.

<table>
<thead>
<tr>
<th>Brick</th>
<th>Vac. saturation porosity $f$</th>
<th>Satiation porosity $f'$</th>
<th>Vac. Satiation porosity $f''$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Golden Purple</td>
<td>0.44</td>
<td>0.25</td>
<td>0.42</td>
</tr>
<tr>
<td>Leicester Buff</td>
<td>0.46</td>
<td>0.29</td>
<td>0.45</td>
</tr>
</tbody>
</table>
The results in table 8.1 show that the vacuum saturation porosity, as expected, was much higher than the satiation porosity for both types of brick. Vacuum saturation porosity represents the saturated state as all the air in the interconnected pore space is forced under pressure to be replaced with water. The satiation porosity on the other hand represents the unsaturated state because only part of the pore space is filled with water by capillary rise. The satiation porosity for both types was about 60% of the vacuum saturation porosity. This suggests that it is not easy to achieve complete saturation and fill all the hidden small pores with fine throats with water by capillary rise. Previous work on clay brick found that it required two years of soaking to reach the same state that vacuum saturation would achieve [124]. However the satiation porosity (f') when measured under vacuum, where there is no air resistance to water capillary rise, gave much higher values than (f') which were almost equal to the vacuum saturation values. This means that the Sharp Front theory assumption of a fully saturated wetted zone agrees with the ideal condition of capillary rise (under vacuum) and not with the reality (laboratory conditions).

Because of the difficulty of poor visibility through the orange colour of the wall of the vacuum tank (transparent clear tanks are not available), this method is extremely difficult to carry out in practice. Confidence in the values obtained is thus less than by the two previous methods.

**8.4.2 Sorptivity**

Sorptivity results measured by the three methods for two samples of Golden Purple brick are shown in figure 8.2 (a), (b) and (c).

Figure 8.2 (a) shows the results of the standard sorptivity method plotted (as i, versus t^{1/2}) measured through the header face for each brick of Golden Purple. The linearity of the relationship was checked by the coefficient of linear correlation which was greater than 0.96. The sorptivity (gradient of the line) is shown on each graph as the coefficient of t^{1/2}.

Figure 8.2 (b) shows the results of sorptivity under vacuum which was very difficult and took a long time to complete. Vacuum sorptivity measured in this way allows us to see if the ideal condition for water movement, where there is no air resistance, is more appropriate to evaluate Sharp Front equations. In this method the wetting front rise faces no pressure as all the air in the pore space is forced out by vacuum action and
therefore the volume of water absorbed with time is much higher than that absorbed in air. The effect is shown from the y axis scale of figure 8.2 (b) compared with 8.2 (a).

Figure 8.2: Variation of $i$ versus $t^{1/2}$ for standard sorptivity in air (a) and under vacuum (b) for Golden Purple bricks, (c) Variation of $x$ versus $t^{1/2}$ for visual method.

Figure 8.2 (c) shows the distance, $x$, that the water rises by capillarity, which linearly increased with $t^{1/2}$. The wet front was not exactly horizontal and this may be due to the existence of cracks and reminds us that the Sharp Front theory although it is useful it is a simplification of reality. The plot of figure 8.2 (c) has two sets of results for the distance $x$, one measured from the right side of the brick sample $x_R$ and the other from the left side $x_L$.

Visual sorptivity was calculated as described earlier in chapter 3 (equation 3.4) from the gradient of the graph of $x$ with $t^{1/2}$ multiplied by the satiation porosity of the brick (table 8.1). The average gradient was used. As an example, the average gradient of sample 1

\[
S = \text{gradient} \times f' , \\
S = 10.25 \times 0.25 = 2.56
\]
is 10.25 mm/min\(^{1/2}\) and of sample 2 is 10.6 mm/min\(^{1/2}\) and the satiation porosity of this brick is 0.25. Therefore \(S_{\text{vis}} = 10.25 \times 0.25 = 2.56\) mm/min\(^{1/2}\) for sample 1 and similarly equal to 2.65 mm/min\(^{1/2}\) for sample 2.

In spite of the difficulties of this visual method, it is a useful illustration of Sharp Front theory. The values of visual sorptivity were close to values from the standard method which gave confidence that this method could have been used to measure sorptivity under vacuum. But for the same reason as for satiation porosity, that is poor visibility through the orange vacuum tank, it was found not to be practical to measure visual sorptivity under vacuum.

Sorptivity results of Leicester Buff brick are presented similarly in figure 8.3.

Figure 8.3: Variation of \(i\) versus \(t^{1/2}\) for standard sorptivity in air (a) and under vacuum (b) for Leicester Buff bricks, (c) Variation of \(x\) versus \(t^{1/2}\) for visual method.
Table 8.2 summarizes the average values of sorptivity of the two types of brick measured by the three methods, given to two decimal places.

Table 8.2: Sorptivity values for the two types of brick.

<table>
<thead>
<tr>
<th>Brick</th>
<th>Sorptivity mm/min&lt;sup&gt;1/2&lt;/sup&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Standard</td>
</tr>
<tr>
<td>Golden Purple</td>
<td>2.87</td>
</tr>
<tr>
<td>Leicester Buff</td>
<td>2.12</td>
</tr>
</tbody>
</table>

Table 8.2 shows that the sorptivity of Golden Purple brick measured in the standard and other methods are higher than that of Leicester Buff brick (this is consistent with chapter 6). For both types of brick the values of vacuum sorptivity were higher than standard sorptivity by approximately 70%. The values of visual sorptivity were close to the standard values, only 10%-15% less.

However the porosity results shown in table 8.1 do not match sorptivity in that the saturated and unsaturated porosity of Golden Purple is less than that of Leicester Buff. This may be related to the pore structure (pore size and distribution) of the brick which contributes substantially to the total porosity and little to capillary suction. Therefore porosity alone cannot be used to predict water movement within a clay brick.

8.4.3 Permeability

Figure 8.4 shows the results from the saturated permeability measurement by constant head method for Golden Purple brick. Figure 8.4 (a) shows the variation of the volume of water collected (mm<sup>3</sup>) with time (min) for four constant hydraulic heads (1000, 1250, 1500 and 1750 mm). The gradient of each line, shown at the side of the graph, is the rate of flow through the sample Q (mm<sup>3</sup>/min). Figure 8.4 (b) shows the variation of Q with hydraulic head H. The linear relationship between flow rate and the hydraulic head clearly confirmed that Darcian flow had occurred through the brick specimens. The value of saturated conductivity K was determined from the gradient of the graph Q versus H and Darcy’s equation (8.2).
Figure 8.4: Results for Golden purple bricks. (a) Volume of water collected versus time and (b) variation of the volume of water per minute versus the hydraulic constant head.

Figure 8.5 shows the permeability results of Leicester Buff bricks in the same way.
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Figure 8.5 Results for Leicester Buff bricks. (a) Volume of water collected versus time and (b) variation of the volume of water per minute versus the hydraulic constant head.

Table 8.3 summarizes the values of saturated conductivity, \( K \), of each brick type, giving each of the two measurements and the average.

Table 8.3: Saturated permeability values for two samples of each type of brick.

<table>
<thead>
<tr>
<th>Brick</th>
<th>( L ) mm</th>
<th>( A ) mm(^2)</th>
<th>( Q/H ) mm(^3)/min</th>
<th>( K ) mm/min</th>
<th>( K ) (Avg.) mm/min</th>
</tr>
</thead>
<tbody>
<tr>
<td>Golden Purple</td>
<td>23</td>
<td>100( \pi )</td>
<td>0.203</td>
<td>0.015</td>
<td>0.014</td>
</tr>
<tr>
<td></td>
<td>21</td>
<td>100( \pi )</td>
<td>0.196</td>
<td>0.013</td>
<td></td>
</tr>
<tr>
<td>Leicester Buff</td>
<td>24</td>
<td>100( \pi )</td>
<td>0.037</td>
<td>0.0028</td>
<td>0.0025</td>
</tr>
<tr>
<td></td>
<td>23</td>
<td>100( \pi )</td>
<td>0.031</td>
<td>0.0022</td>
<td></td>
</tr>
</tbody>
</table>

It can be seen from table 8.3 that the permeability values are reproducible. There is a strong difference in permeability between the two bricks, whereas the difference in their
open porosities is small (table 8.1). Permeability in ceramic materials depends on the size and distribution of the pores and the amount of less complicated and more permeable paths for water flow. It is generally believed that the complex microstructure of porous construction materials, which may include microcracks and in some cases compounds which react with water, controls the hydraulic conductivity. Permeability of fired clay bricks depends also on other factors such as the degree of firing [127]. It is worth noting that the permeability results of this study are consistent with those reported by other workers [7, 23, 128]. Hall and Hoff [7] found saturated permeability for clay brick ceramic, whose vacuum saturation porosity was 0.4, to be 0.00192 mm/min and Ioannis et al. [23] reported a permeability of 0.00190 mm/min for a clay brick of vacuum saturation porosity 0.41. This agreement with the results of other workers which were obtained using a different method of high pressure permeameter (Hassler cell) besides the good reproducibility in results gives confidence in the reliability of this simple method and its practical importance.

It is necessary to say that measuring the permeability of construction materials is much more difficult than measuring sorptivity or porosity. Further, the measurement of unsaturated conductivity is not practicable because it requires maintaining the sample in the same state of unsaturation while supplying the water at the inflow face [24].

### 8.4.4 Capillary pressure

In order to get an idea of the time needed for the wetting front to reach the top of an unsealed specimen by capillary rise, equation 8.7 (equation 6.5 in chapter 6) given by Hall and Hoff [7] was used

\[ t = \left( \frac{f' x}{S} \right)^2. \]  

(8.7)

The time required for capillary rise to travel through 160 mm of Golden Purple brick (satiation porosity 0.25 and sorptivity 2.87 mm/min\(^{1/2}\)) was about 4 hours and to travel 160 mm of Leicester Buff brick (satiation porosity 0.29 and sorptivity 2.12 mm/min\(^{1/2}\)) was about 9 hours. These calculations are for absorptions where there is free escape of displaced air, and the time taken for that length of brick represents a minimum not an optimum value. However in the case of absorption into a sealed sample (as in this work) the wetting front advances faces resistance from the compressed air in front of it and therefore it takes longer time to reach the top of specimen. Absorption stops when the pressure of the trapped air equals the wetting front capillary pressure [7].
Figures 8.6 and 8.7 show the variation of water absorption with time obtained in the initial experiments for two specimens of each type of brick.

Figure 8.6: Main graph, variation of mass of absorbed water with time measured from immersion time for two samples of Golden Purple bricks, GP1 and GP2 over 2000 hours. Inset graph shows the same variation over 200 hours.

Figure 8.7: Main graph, variation of mass of absorbed water with time measured from immersion time for two samples of Leicester Buff bricks, LB1 and LB2 over 2000 hours. Inset graph shows the same variation over 200 hours.

These graphs show the rate of absorption for a period of nearly 3 months (2000 hours). The increase in the mass of the samples was measured daily. It can be seen that the
rapid rate of absorption happened within the first days. An inset graph has been drawn in each figure with a smaller scale for the $x$ axis (200 h) to show more clearly the time that the absorption slows down and the graph becomes nearly horizontal.

For Golden Purple brick the inset graph shows the absorption rate was rapid during the first 24 hours then continued in a very slow rate, whereas the inset graph of Leicester Buff brick demonstrates clearly that the absorption rate was continued rapidly for 50 hours before it became slower. After 50 hours the absorption for both types was very slow but it never stopped when monitored for nearly 3 months. The reason for this may be due to the existence of a very small leakage in the epoxy sealing which is unavoidable in practice and led to the escape of air. The other explanation for this is that the high-pressure trapped air gradually dissolves in the wetted zone, thus allowing more water to be absorbed and the graph will never become horizontal. This will change the state of the brick gradually from satiation to saturation.

Figure 8.8 shows the variation of mass of absorbed water (average of two) with time (200 h) for the two types of brick plotted on the same graph.

![Figure 8.8: Variation of mass of absorbed water (average of two) with time in 200 hours for both types of brick. GP=Golden Purple; LB=Leicester Buff.](image)

It can be seen more clearly that the absorption rate of Leicester Buff brick is slower than Golden purple brick, and that early age data (within first 50 h), which are important, are missing. For Golden Purple brick the time considered for completed water rise (average of two) to be around 24 h and about 50g of water absorbed whereas the time needed to complete water rise for LB brick is around 50 h and about 45 g of water absorbed.
The calculations of capillary pressure $\Psi$ obtained from these initial experiments for each sample are presented in table 8.4. The inset of figures 8.6 and 8.7 are used to consider the mass increase at the beginning of the horizontal part of each graph. This mass increase gave the volume of water absorbed which determined the volume of pores filled with water by capillary rise.

For Golden Purple brick the mass increase was taken at time 24 h as 50.96 g and 48.92 g for sample 1 and 2 respectively while the mass increase for Leicester Buff brick was taken at time of 50 h as 49.86 g and 43.97 g for sample 1 and 2 respectively. Mass increase is weighed to ± 0.01 g which means that the volume of capillary rise is measured to ± 0.01 cm$^3$ or 10 mm$^3$. It is very encouraging that these initial experiments have given an idea of the error in volume of capillary rise. Therefore the right order of magnitude of capillary rise is four significant figures.

Porosity is taken from table 8.1. Vacuum saturation porosity $f$ is used because it measures the available volume of pore space.

Table 8.4: Capillary pressure measurements of the initial experiments for the two types of brick.

<table>
<thead>
<tr>
<th>Brick</th>
<th>Prism dimensions (mm)</th>
<th>Vol. of pore space (prism vol.*$f$, $v_1$) (mm$^3$)</th>
<th>Vol. of capillary rise (mass increase *1000) ($v_2$) (mm$^3$)</th>
<th>Vol. of trapped air ($v_3$) (mm$^3$)</th>
<th>Capillary pressure $p_2$ (MPa)</th>
<th>Mean value of capillary pressure (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GP</td>
<td>160<em>39</em>30</td>
<td>82368</td>
<td>50960</td>
<td>31408</td>
<td>0.164</td>
<td>0.158</td>
</tr>
<tr>
<td></td>
<td>160<em>40</em>29</td>
<td>81664</td>
<td>48920</td>
<td>32744</td>
<td>0.151</td>
<td></td>
</tr>
<tr>
<td>LB</td>
<td>160<em>40</em>32</td>
<td>94208</td>
<td>49860</td>
<td>44348</td>
<td>0.114</td>
<td>0.115</td>
</tr>
<tr>
<td></td>
<td>155<em>40</em>29</td>
<td>82708</td>
<td>43970</td>
<td>38738</td>
<td>0.115</td>
<td></td>
</tr>
</tbody>
</table>

For Golden Purple brick and using the ideal gas equation (8.4)

$v_1 =$ prism volume*vacuum saturation porosity
$v_1 = (160*39*30)*0.44 = 82368 \text{ mm}^3$

$v_2 =$ volume of capillary rise
$v_2 = 82368 - 50960 = 31408 \text{ mm}^3$. $p_1 =$ atmospheric pressure $= 0.101325 \text{ MPa}$ and for this study it is approximated to three decimal places (0.101)

Substituting the values of $v_1$, $v_2$ and $p_1$ into equation 8.4 to calculate $p_2$ gives

$p_2 = \frac{p_1 v_1}{v_2}$ (8.4)
So that \( p_2 = 0.101 \times 82368/31408 = 0.265 \text{ MPa} \)

The wetting front capillary pressure = pressure of displaced air - atmospheric pressure

\[ = 0.265 \text{ MPa} - 0.101 \text{ MPa} = 0.164 \text{ MPa}. \]

These results show how air is displaced and trapped during the absorption of water by these types of clay bricks. In the wetted region behind the advancing front, the volume of capillary rise filled about 50-60% of the total open porosity. The remaining pore space is filled with trapped air.

These initial experiments did not give the early-time data clearly and there are not enough data points during the early hours of absorption. Therefore the experiment was repeated and the mass of absorbed water was collected at constant time intervals of 10 minutes from immersion by using an electronic balance which was connected to a computer as explained earlier in section 8.3.5. Figure 8.9 shows the variation of mass of water absorbed with time in the 50 hours of absorption for both types of brick.

![Figure 8.9: Variation of mass of absorbed water with time over 50 hours of immersion for both types of brick. GP = Golden Purple; LB = Leicester Buff.](image)

It can be seen that there are so many data points that it is easier to see when the graph has become horizontal. Even with no leak in the coating the author believes that, given accurate mass measurement methods, the graph will never become horizontal due to the continued dissolution of high-pressure trapped air. The initial rate of absorption of Leicester Buff brick is lower than that of Golden Purple brick. The final amount of water absorbed by capillarity depends on porosity rather than suction. As the lengths of these samples (70 mm) are less than half the length of the initial experiment samples.
(160 mm), therefore the mass increase of absorbed water and the time needed for the graph to be horizontal (completion of absorption) are about half the initial samples. For Golden Purple brick the completion of absorption took after about 10 h with mass increase of 20 g while Leicester Buff brick took about 30 h to complete absorption with mass increase of 21.5 g as shown in figure 8.9.

The capillary pressure $\Psi$ was calculated in the same way from the volume of absorbed water using the ideal gas equation.

Table 8.5: Capillary pressure measurements of the refined method.

<table>
<thead>
<tr>
<th>Brick</th>
<th>Prism dimensions (mm)</th>
<th>Vol. of pores ($v_1$, mm$^3$)</th>
<th>Vol. of capillary rise (mass increase *1000, mm$^3$)</th>
<th>Vol. of trapped air ($v_2$, mm$^3$)</th>
<th>Capillary pressure $p_2$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GP</td>
<td>70<em>40</em>30</td>
<td>36960</td>
<td>20000</td>
<td>16960</td>
<td>0.119</td>
</tr>
<tr>
<td>LB</td>
<td>70<em>40</em>29</td>
<td>37352</td>
<td>21500</td>
<td>15852</td>
<td>0.137</td>
</tr>
</tbody>
</table>

These measured values of capillary pressure from the refined experiment are different and more accurate than the initial values. These results are consistent with the results of previous work who suggest that capillary pressure relates to the saturated permeability in addition to pore diameter and shape [128]. The capillary pressure of Golden Purple brick is 0.119 MPa (saturated permeability 0.014 mm/min) which is lower than that of Leicester Buff brick 0.137 MPa whose saturated permeability is 0.0025 mm/min.

The values of measured capillary pressure of both experiments have good agreement with the results of previous works on clay brick [23, 128].

The capillary pressure values of the refined experiments are taken in the validation of the Sharp Front equation in the next section.

### 8.5 VALIDATING THE EQUATION $S = (2Kf\Psi)^{1/2}$

Having measured all the hydraulic parameters on the same two samples of brick, Table 8.6 summarises the values of the experimental results, (mean of 2) for the two types.
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Table 8.6: Summary of the experimentally determined values of sorptivity, porosity, permeability and capillary pressure (mean of 2).

<table>
<thead>
<tr>
<th>Property</th>
<th>Golden Purple brick</th>
<th>Leicester Buff brick</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Sorptivity</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Standard sorptivity $S$ (mm/min$^{1/2}$)</td>
<td>2.87</td>
<td>2.12</td>
</tr>
<tr>
<td>2. Visual sorptivity $S_{vis}$ (mm/min$^{1/2}$)</td>
<td>2.61</td>
<td>1.95</td>
</tr>
<tr>
<td>3. Vacuum sorptivity $S_{vac}$ (mm/min$^{1/2}$)</td>
<td>4.65</td>
<td>3.54</td>
</tr>
<tr>
<td><strong>Porosity</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1. Vacuum saturation porosity $f$</td>
<td>0.44</td>
<td>0.46</td>
</tr>
<tr>
<td>2. Satiation porosity $f'$</td>
<td>0.25</td>
<td>0.29</td>
</tr>
<tr>
<td>3. Vacuum satiation $f''$</td>
<td>0.42</td>
<td>0.45</td>
</tr>
<tr>
<td><strong>Permeability</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Permeability $K$ (mm/min)</td>
<td>0.014</td>
<td>0.0025</td>
</tr>
<tr>
<td><strong>Capillary pressure</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Measured capillary pressure $\Psi$ (MPa)</td>
<td>0.119</td>
<td>0.137</td>
</tr>
</tbody>
</table>

The experimental results that are used in the verification of equation 8.1 are presented in table 8.7. Visual sorptivity is not included because there is less confidence in the results and they do not give any more information than given by the standard method. A similar consideration applies to the vacuum satiation porosity $f''$: it is almost identical to the vacuum saturation porosity.

Table 8.7: Summary of the experimentally determined values of $S$, $f$, $K$ and $\Psi$ used in the verification of equation 8.1.

<table>
<thead>
<tr>
<th>Measured property (Average value)</th>
<th>Golden Purple brick</th>
<th>Leicester Buff Brick</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard sorptivity $S$ (mm/min$^{1/2}$)</td>
<td>2.87</td>
<td>2.12</td>
</tr>
<tr>
<td>Vacuum sorptivity $S_{vac}$ (mm/min$^{1/2}$)</td>
<td>4.65</td>
<td>3.54</td>
</tr>
<tr>
<td>Vacuum saturation porosity $f$</td>
<td>0.44</td>
<td>0.46</td>
</tr>
<tr>
<td>Satiation porosity $f'$</td>
<td>0.25</td>
<td>0.29</td>
</tr>
<tr>
<td>Permeability $K$ (mm/min)</td>
<td>0.014</td>
<td>0.0025</td>
</tr>
<tr>
<td>Measured Capillary pressure $\Psi$ (MPa)</td>
<td>0.119</td>
<td>0.137</td>
</tr>
</tbody>
</table>
Based on the assumption of the Sharp Front theory the equation considered is

\[ S = (2Kf\Psi)^{1/2}. \]  

(8.1)

Squaring equation 8.1 gives

\[ S^2 = 2Kf\Psi. \]  

(8.6)

Rearranging equation 8.6 gives

\[ \Psi = \frac{S^2}{2Kf}. \]  

(8.8)

From equation (8.8) the wetting front capillary pressure \( \Psi \) can be calculated from the experimental values of sorptivity, porosity and saturated hydraulic conductivity. Capillary pressure was determined in mm of water and in the equivalent MPa. The unit MPa is equal to the pressure of 101971.6213 mm of water at 4°C [129]. For the purpose of the calculations of this study 1 MPa is approximated as 100000 mm of water vapour pressure. If the calculated and measured values are close, this is taken as validation of equation 8.1.

An example calculation is now presented.

The calculated capillary pressure for Golden Purple brick using standard sorptivity and vacuum saturation porosity is:

\[ \frac{S^2}{2Kf} = \frac{(2.87)^2}{2 \times 0.014 \times 0.44} = 668.58 \text{ mm}. \]

This value is equivalent to 0.007 MPa, taking the value of 1 MPa = \( 10^5 \) mm of water.

This capillary pressure should include atmospheric pressure (0.101 MPa). Therefore the calculated value of \( \Psi \) is 0.108 MPa for Golden Purple brick.

Table 8.8 shows the calculated values of capillary pressure using both standard and vacuum sorptivity and both vacuum saturation and satiation porosity values already presented in table 8.7.
Table 8.8: Calculated values of the capillary pressure in mm and MPa for the Golden Purple (GP) and Leicester Buff (LB).

<table>
<thead>
<tr>
<th>Brick</th>
<th>$S$ (mm/min$^{1/2}$)</th>
<th>$f$</th>
<th>$K$ (mm/min)</th>
<th>Calculated capillary pressure (mm)</th>
<th>Calculated capillary pressure (MPa)</th>
<th>Calculated Capillary pressure + atmospheric pressure $\Psi$ (MPa)</th>
<th>Measured Capillary pressure ($\Psi$) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>GP</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.87</td>
<td>0.44 (vac. saturation)</td>
<td>0.014</td>
<td>668.58</td>
<td>0.007</td>
<td>0.108</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4.65</td>
<td>0.44 (vac. saturation)</td>
<td></td>
<td>1755.07</td>
<td>0.018</td>
<td>0.119</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.87</td>
<td>0.25 (air satiation)</td>
<td></td>
<td>1176.70</td>
<td>0.012</td>
<td>0.113</td>
<td>0.119</td>
<td></td>
</tr>
<tr>
<td>4.65</td>
<td>0.25 (air satiation)</td>
<td></td>
<td>3088.93</td>
<td>0.031</td>
<td>0.132</td>
<td></td>
<td></td>
</tr>
<tr>
<td>LB</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>2.12</td>
<td>0.46 (vac. saturation)</td>
<td>0.0025</td>
<td>1954.09</td>
<td>0.019</td>
<td>0.121</td>
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<tr>
<td>3.54</td>
<td>0.46 (vac. saturation)</td>
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<td>5448.52</td>
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<td>0.155</td>
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<td>0.29 (air satiation)</td>
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<td>3099.59</td>
<td>0.031</td>
<td>0.132</td>
<td>0.137</td>
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<td>3.54</td>
<td>0.29 (air satiation)</td>
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<td>8642.48</td>
<td>0.086</td>
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</table>

The measured capillary pressure value is obtained in the unsaturated state (standard sorptivity and satiation porosity). Comparing with the calculated value of the same state for both types of brick (fourth and eighth row of table 8.8), it can be seen that they are very close. This closeness between the measured and calculated value validates the Sharp Front equation. It can be seen that the vacuum sorptivity (which is 70% higher than standard sorptivity) gives higher capillary pressure with both values of porosity. The vacuum saturation porosity (which is 60% higher than satiation porosity) gives lower capillary pressure with both values of sorptivity. This is because $\Psi$ is directly proportional to $S^2$ and inversely proportional to $f$ as shown in equation (8.8).

These results confirm that the Sharp Front equation is subject to many assumptions. Sorptivity is measured on completely dry porous solid materials. The wetted zone in capillary rise is assumed to be fully saturated which means all the air which was present in the open porosity is displaced. The Sharp Front model equation also uses the saturated hydraulic conductivity.
It is worth noting that the calculated and measured values are both between 0.1-0.2 MPa which is the range of capillary pressure for common clay bricks highlighted by Ioannou et al. [23].

8.6 CONCLUSIONS

- Validation of the fundamental equation experimentally is not straightforward. It is difficult and time-consuming. Sharp Front theory gives a sound and sensible approximation for a variety of water transport processes occurring in fired clay bricks and other porous materials.
- The experimental results of this study have been shown to give good agreement with Sharp Front models. The Sharp Front model is a valid theory for porous solids provided they are unreactive with water. Clay brick is an ideal material to work with for this reason.
- Determination of capillary pressure for clay brick materials is very challenging because of the difficulties with sample preparation. The experimental method used in measuring the equilibrium pressure of displaced air is very important because it provides a simple means of measuring directly the wetting front capillary pressure which needs to be used to dewater a mix to the same extent that it would be dewatered in practice.
- Porosity alone cannot be used to predict the sorptivity or water movement in porous materials. Values of vacuum saturation porosity are almost equal to the vacuum saturation porosity because there is no air resistance to capillary rise.
- Sorptivity can be measured by visual inspection of the wet front rather than by weighing. Although the wet front rise may not be horizontal the results of the visual method are close to the sorptivity determined by the standard method.
- Standard sorptivity can also be measured under vacuum although it is a very difficult and lengthy procedure. Vacuum sorptivity values for clay bricks were 70% higher than standard sorptivity values which demonstrates clearly the effect of air pressure on capillary rise.
- Permeability of clay bricks can be measured by the simple constant head method provided that a well sealed sample is used with a constant vertical head of water.
- It is easier to measure $\Psi$ directly than to calculate it from measurement of its defining parameters.
CHAPTER 9  CONCLUSIONS AND SUGGESTIONS FOR FUTURE WORK

9.1 MAJOR CONCLUSIONS

From the literature review it has been shown that there are a large number of journal articles on fresh mortars but it is extremely difficult to find published work on dewatered mortars. The dewatered state is an ignored area of study although dewatering of freshly mixed mortar by an absorbent substrate has great effect on the fresh and hardened properties of mortar.

It has been demonstrated that the API pressure cell technique is the best available method for measuring the desorptivity of freshly mixed mortars because it can safely apply the pressure which is equivalent to the capillary pressure of the highly absorbent substrates such as brick and block. However the API dewatering is not uniform along the length of the resultant filter cake and it is only about 50% of the complete dewatering by a masonry substrate. The precision of the API pressure cell test is reasonably high according to the statistical analysis of repeatability standard deviation.

It is shown that lime-based mortars possess high water retentivity in comparison with cement-based mortars but it is important, for experimental investigation purposes, that they all come from the same provider.

This study has shown that it is possible to examine the effect of addition materials on the water retaining behaviour of freshly mixed hydraulic mortars in a quantitative way by measuring the desorptivity. The addition of 20% of non-pozzolanic limestone powder slightly reduces the desorptivity of CEM I Portland cement. In contrast the presence of 50% by volume of the pozzolanic addition GGBS increases the desorptivity of NHL5 by nearly 25%. Surprisingly the results of this study indicate that particle size distribution of the binder material has little effect on desorptivity when compared to the effect of hydraulicity. On the other hand adding sulphate ions to the mix water of hydraulic lime mortars (with and without GGBS addition) increases their desorptivity.

The results of this investigation have confirmed that the 1 minute absorption test can give misleading results and sorptivity is seen as a more accurate comparison of water
absorption ability. Common clay bricks are anisotropic in water absorption and transmission and in general the sorptivity of extruded engineering brick is very much lower than pressed brick.

The results of the developed sorptivity method (visual sorptivity) are close to those determined by the standard method yet vacuum sorptivity values were 70% higher than standard sorptivity. This is because there is no air resistance to capillary rise.

The time for full capillary absorption by immersion is different for different brick types. Therefore the immersion time needs to be selected carefully in the test procedures and standards. The water absorption standard BS EN 772-21:2011 needs to be revised and improved.

The satiated porosity is generally much less than the vacuum saturation porosity but when it is measured under vacuum it gives almost equal results to vacuum saturation values. It is worth saying that the vacuum satiation method was easy to carry out but it was not possible to obtain precise results because of the opaque vacuum chamber. This difficulty could be overcome in the future with the availability of transparent clear vacuum tanks.

It is also concluded that porosity alone does not predict or explain water movement within brick. For instance the porosity of Leicester Buff bricks is higher than that of Golden Purple bricks, although the sorptivity of the latter is higher.

This study has shown that it is possible to dewater mortars to the same extent that they will be dewatered in use by a suitably designed and constructed brick mould.

The initial and final setting times of cement and hydraulic lime mortars are greatly reduced by dewatering and both flexural and compressive strength increased significantly. The sorptivity of the set mortar is greatly reduced by dewatering in the freshly mixed state. These results suggest that the standard steel mould used in testing the setting time or strength of mortars gives values that underestimate the time to set and the strength of mortars in masonry. Therefore there are implications for standards BS EN 196-1:2005, BS EN 196-3:2005 and BS EN 1015-11:1999.
Although simple in principle, measuring the capillary pressure of a clay brick is not easy because of the difficulties with sample preparation. The experimental technique used in measuring the equilibrium pressure of displaced air is crucial because it provides a simple means of measuring directly the wetting front capillary pressure which needs to be used to dewater a mix to the same extent that it would be dewatered in practice.

It has been shown that the validation of the Sharp Front fundamental equation experimentally is not straightforward. It is difficult and time consuming. The experimental results suggest that the Sharp Front model is a valid theory for porous solids provided they are unreactive with water. Clay brick is an ideal material to work with for this reason.

The work of this thesis has led to three published papers. These papers are attached in appendix B.

**9.2 SUGGESTIONS FOR FUTURE WORK**

This research work considered some of the dewatering issues, however some other important questions which have not been addressed are:

- A detailed experimental investigation of dewatering extended into concrete studies.
- Possibility of further development of pressure cell on slurry other than building mortars. The results of this study should assist in finding and recommending a valuable standard test method for measuring the water retaining properties of mortars.
- A further detailed study of how saturated permeability varies with time for set and hardened mortars that have been cast in both steel and absorbent moulds. This is important for masonry durability and may have further implications for standards.
- A detailed study of the effect of dewatering on the properties of layered mortars would be interesting. This study would have practical applications to mortars used in plastering and rendering where successive layers are applied and dewatered in turn by the preceding layer.
• A detailed study of the microstructure of both dewatered and non-dewatered hydraulic mortars by Scanning Electron Microscopy (SEM). The SEM images could be used to examine the effect of dewatering on the microstructure of mortars.

• Experimental and theoretical approaches to masonry mortars should be further developed, possibly to include problems associated with damp penetration in structures.
## REFERENCES


[96] British Standards Institution BS EN 772-11: 2011, Methods of test for masonry units: Determination of water absorption of aggregate concrete, autoclaved aerated concrete, manufactured stone and natural stone masonry units due to capillary action and the initial rate of water absorption of clay masonry units. 2011: Milton Keynes:BSI.


# APPENDIX A  CALCULATIONS FOR REPEATABILITY STANDARD DEVIATION

1. One-second time interval

<table>
<thead>
<tr>
<th>Time (s)</th>
<th>Desorbed water mass (g)</th>
<th>Mean (of 3)</th>
<th>Standard deviation $S_1$</th>
<th>$S_1^2$</th>
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<td>0.01</td>
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<td>0.44</td>
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</table>
Appendix A: Calculations for repeatability standard deviation

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<table>
<thead>
<tr>
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</table>

$S_r = \sqrt{\frac{\sum S_i^2}{p}}$

$S_r = \sqrt{\frac{34.80}{39}} = 0.944 \approx 0.94$ g/mi
Appendix A: Calculations for repeatability standard deviation

2. Two-seconds time interval

<table>
<thead>
<tr>
<th>Time (s)</th>
<th>Desorbed water mass (g)</th>
<th>Mean (of 2)</th>
<th>Standard deviation $S_i$</th>
<th>$S_i^2$</th>
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\[ S_r = \sqrt{\frac{0.42}{17}} = 0.157 \equiv 0.16 \text{ g/min} \]
Appendix A: Calculations for repeatability standard deviation

3. Three-seconds time interval

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\[ S_r = \sqrt{\frac{1.43}{14}} = 0.319 \equiv 0.32 \text{ g/min} \]

4. Ten-seconds time interval

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\[ S_r = \sqrt{\frac{0.91}{4}} = 0.477 \equiv 0.48 \text{ g/min} \]
APPENDIX B  CONFERENCES PAPERS

Young researchers' forum in construction materials
17 May 2012
Society of chemical industry

THE EFFECT OF DEWATERING ON STRENGTH AND SETTING TIME OF HYDRAULIC MORTAR

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M13 9PL, UK. * (nidhal.aldefai@postgrad.manchester.ac.uk)

1. Introduction

In masonry construction jointing mortars, renders and plasters are applied in the wet state in thin
layers to absorbent substrates such as brick and are dewatered before the setting process begins.
The absorption of water from fresh mortar by clay brick depends on both the sorptivity of the
substrate and desorptivity of the fresh mix [1]. Many researchers [1-3] have shown that lime based
mortars possess high water retaining ability in comparison with cement based mortars.
Water retention is the ability of a fresh mortar to retain its mix water when placed in contact with an
absorbent substrate. In 2002, Hall and Hoff [4] derived the theoretical basis and quantified the water
retaining ability of fresh mortars by a well-defined parameter, the desorptivity $R_d$, obtained from the
gradient of a plot of volume of water absorbed per unit area versus the square root of time.
The present work reports an investigation of the effect on mortars of dewatering by clay brick using
an absorbent mould of the same dimensions as the standard steel test mould (40x40x160 mm). The
properties examined are initial and final setting time; and compressive and flexural strength.

2. Mould design

Calculations were carried out to determine the thickness of brick and the time required to dewater a
wet mortar mix in a brick mould of the same dimensions as a standard steel mould. A theoretical
model which defines the time to dewater a freshly mixed mortar is described in [5]. Since the mould
is constructed of sufficient thickness of brick, the wet mortar is fully dewatered prior to setting.
Golden purple brick was used for the mould construction as it has relatively high sorptivity. Figure 1
shows the brick mould and a schematic representation of the flow of mix water from the wet mix to
the absorbent mould.

(a) 

(b)

Figure 1: (a) the brick mould, (b) schematic representation of dewatering.
3. Experimental work

The binder materials used in the experimental work were CEM1 Portland cement and St Astier natural hydraulic lime NHL5. NHL5 was chosen because it is the most hydraulic of the natural hydraulic limes and its setting is mainly by hydration reactions with the carbonation reaction having less effect. Mortar sand was dried at 105 °C, and the sieved fraction of 300-600 μm was used in all mixes. A single mix design of 1: 2: 0.78 (binder: sand: water) by volume was examined in this study. This mix proportion is recommended by the manufacturer to give optimum workability [1]. The mass of both binder and sand required to produce the mix proportions by volume were calculated after careful determination of density values. To ensure consistency, a standard mixing regime was used. Initial and final setting times were determined using the Vicat method (BS EN 196-3:2005 and A1:2008). The initial setting time is defined as the time elapsed since the mixing water was added to the binder to when the needle penetration is 4 mm from the base of the mould. The final setting time is defined as the time when there is less than 0.5 mm penetration of the needle. Setting time was determined for the dewatered and non-dewatered cement and lime mortars at two different temperatures: 15°C and 25°C. For the dewatered mortars, Vicat measurements were not begun until the wet mix had been fully dewatered. This time was established by calculations.

Flexural strength measurements were carried out according to BS EN 196-1: 2005. Six prism samples of each mortar mix at each age were tested: three dewatered samples (brick mould) and three non-dewatered (steel mould). Following the flexural test, each bar produced two samples which were then used for compressive strength measurement. The compressive strength was measured on one of these two pieces.

4. Results and discussion

Comparison of the results obtained from the Vicat test for initial and final setting time of dewatered and non-dewatered cement and hydraulic lime mortars at 15 and 25°C are shown in figure 2. The results show clearly that the lime initial and final setting times are longer than for cement. This is because the initial setting time is the time needed to change from a fluid to a rigid stage [6], and the final setting time represents the end of workability and the beginning of hardening. Setting of hydraulic lime is slower than cement mortar because the dominant calcium silicate present in hydraulic lime is C₃S and not C₃S. Also, both initial and final setting times are reduced for both by dewatering. The initial setting time of dewatered cement mortar at 15°C was reduced from 3.92 h to 0.75 h which represents an 80% reduction. The final setting time was reduced by a similar amount: from 6.2 h to 2.4h and represents 61%. The dewatering effect on setting time of lime mortar is nearly the same. The initial and final setting of NHL5 mortar were reduced by approximately 6 hours at 15°C which represents 77% and 61% reduction of the initial and final setting time respectively. Also it is well known that setting time is affected by temperature, it reduces as the temperature rises. The results in figure 2 also show that the reduction in setting time of dewatered CEM I and NHL5 mortars is greater at 25°C than at 15°C. Although the Vicat test does not give an obvious link to hydration and it is not a fundamental measure of setting time, the test is, however, entirely suitable for the comparative results carried out in this work. It is worth noting that in the development of new more sophisticated setting time test methods [7-9], the fundamental issue that the dewatered mortars are different to the non-dewatered mortars is not addressed.

The strength results presented in figure 3 show the average values (of three) of flexural (f₀) and compressive strength (fₓ) of both dewatered and non-dewatered lime and cement mortars at 7, 28, 56 and 90 days. Dewatered samples show a higher strength at all ages than the non-dewatered samples of cement and lime mortars. In each case the strength is increased following dewatering in the wet-state. The higher increase in both compressive and flexural strength of dewatered cement samples is at 7 days (37% and 27% respectively. Figure 3 (a) & (b)). For NHL5 flexural strength of dewatered samples at all ages is 3-4 times the non-dewatered as shown in figure 3 (c). The compressive strength of dewatered NHL5 samples at 28 days is more than 3 times that of non-
dewatered samples and at 56 days is more than 4 times but shows little further increase thereafter as shown in figure 3 (d). The water retentivity of Portland cement mortar is much less than that of natural hydraulic lime NHL5 mortar [1-3], therefore the loss of water from cement mortar on dewatering is higher than the loss from the equivalent NHL5 mortar. The strength of cement at early age (7 days) increased by this abstraction of mix water therefore the difference is higher for the cement mortars at early age.

Figure 2: Initial and final setting time for dewatered and as-mixed (non-dewatered) mortars at 15 and 25°C. CEM I mortars (a) & (b), and NHL5 mortars (c) & (d).

Figure 3: Flexural and compressive strength of dewatered and non-dewatered mortars. CEM I mortars (a) & (b), and NHL5 mortars (c) & (d). (Note y axis scales.)
5. Conclusions

- It is possible to dewater mortars to the same extent that they will be dewatered in use by a suitably designed and constructed brick mould.
- The initial and final setting times of both cement and hydraulic lime mortars are greatly reduced by dewatering.
- Dewatering results in a significant increase in both flexural and compressive strength.
- The higher the desorptivity, $R$, the more the water releasing the mortar and therefore the greater the effect of dewatering on both setting time and strength.
- The standard steel mould used in testing the strength of mortars gives values that underestimate the strength of mortars in masonry. Therefore there are implications for standards: mould material, procedure, and time to testing should all be considered. Currently lime mortars are tested at 28 days as for cement mortars, and clearly this time requires consideration.

6. REFERENCES


Appendix B: Conferences papers

The influence of pozzolanic additions on the water retentivity of freshly-mixed hydraulic lime mortars

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School of Mechanical, Aerospace and Civil Engineering,
The University of Manchester, Manchester, UK

ABSTRACT

The purpose of this paper is to examine the effects of hydraulicity, binder particle size and addition of the pozzolanic ground granulated blast furnace slag (GGBS) on the desorptivity of mortars made with natural hydraulic lime (NHL5) and to compare these with Portland cement (CEM I) mortar. It is shown that CEM I mortar is more water releasing than NHL5 mortar. This is to be expected as CEM I (being PC-based) is the more hydraulic. In this study of replacement of the NHL5 binder with varying volume fractions of GGBS, it is shown that replacement levels up to 50% have little effect on the desorptivity, but that increasing levels from 50 to 100% produces a systematic increase in desorptivity. This result is attributable to two factors: increased hydraulicity of the NHL mortar caused by the pozzolanic addition, and the increased average particle size of the composite binder caused by the coarser GGBS. This suggests the possibility of manipulating the desorptivity of NHL mortars.

1. INTRODUCTION

In construction, mortars are used in bricklaying and rendering. In these applications mortars are applied as a wet mix in thin layers to absorbent substrates such as brick, stone and concrete and dewatered by the capillary suction of these substrates before the beginning of setting. The mix water abstraction from fresh mortar by the substrate depends on both the suction of the substrate and the desorptivity of the fresh mix (Collier et al., 2007). The suction of the substrate and the water retentivity of the wet mix are in competition. This has significant influence both on fresh mortar properties of workability and setting time (Beall, 2003) and the resultant hardened mortar properties such as adhesion, strength and durability. The use of high suction bricks with low water retaining mortars may lead to insufficient hydration of the hydraulic binder and thus a decrease in mechanical strength (Sébabi et al., 2003). Low suction materials typical of engineering bricks will abstract very little water, and the excess in the mix will reduce the bonding capacity of the mortar to the brick (Robinson, 1996). Durability may also be adversely affected. Therefore mortars of low water retentivity are more suitable for use with low suction bricks and vice versa.

Sorptivity is defined in a very simple way (Gummerson et al., 1980) as the tendency of a porous material to absorb and transmit water or other wetting fluid by capillarity. Hall and Hoff derived the theoretical basis for and quantified the water retaining ability of freshly mixed wet mortars by a well-defined parameter, the desorptivity \( R \) (Hall and Hoff, 2002). This is obtained from the gradient of a plot of the desorbed volume of water / unit area versus the square root of time. The water retaining ability of fresh mortars can be measured using an American Petroleum Institute (API) pressure cell. Many researchers (Carter et al., 2003; Collier et al., 2007; Green et al., 1999) have determined the desorptivity of cement and lime mortars and have shown that desorptivity depends greatly on the hydraulicity of the binder material used in the mortar mix. Hydraulicity, defined as the ability of the material to set under water, is determined by the amount of clay impurity in the original limestone. The hydraulicity of binders, in order of most to least, is Portland cement, hydraulic limes (NHL5>NHL3.5>NHL2) and hydrated lime. It has been shown that lime-based mortars possess high water retentivity in comparison with cement-based mortars, but there is little explanation of the reasons for this. Hendrickx et al. (2008) found that the higher the particle surface area of a lime mortar, the higher the water retentivity. Ince et al. (2010) showed that a decrease in sand particle size caused a progressive decrease in desorptivity of mortars in general. This present work investigates for the first time the effect of the binder particle size on the mortar desorptivity.

There are many types of industrial pozzolanic waste products which can be used as cement replacement materials to improve performance or to reduce the energy of production (Erakdi et al., 2006). Among these fly ash (FA), silica fume (SF) and ground granulated blast furnace slag (GGBS) are the most widely used. Additions of these materials to concrete result in improve
Appendix B: Conferences papers

compressive strength and durability. The same pozzolanic materials may also be added to lime mortars. The addition of pozzolanic material to lime mortar changes its properties: density and strength increase while porosity reduces depending on the type of pozzolan used (Gibbons, 1997). In mortars it is common practice to add lime to Portland cement in order to improve the early age rheological properties such as water retention and workability. However once setting and hardening has begun, compressive strength becomes the main property required and lime has little role to play because of its low strength. Therefore the replacement of some of the lime with pozzolanic material has been shown to be a great advantage in increasing the strength of lime mortars (Fragata et al., 2007; Taha and Shrive, 2001).

The purpose of this paper is to demonstrate the effects of the addition of the pozzolanic material GGBS on the water retaining properties of freshly mixed lime mortars. The role of both hydraulicity and binder particle size on the water retentivity of freshly mixed mortars are reported. A range of volume fraction additions of GGBS were added to NHL5 and the effects of these on the desorptivity were examined. The particle size of eight binder materials: CEM I, NHL5, NHL5-Z, NHL3.5, GGBS as well as mixes of NHL5 + 50% GGBS, NHL5 + 75% GGBS and NHL3.5 + 10% GGBS were measured and the effects on desorptivity examined. The effect of applied gas pressure on desorptivity is also reported.

2. EXPERIMENTAL DETAILS

2.1 Materials and mix design

The hydraulic limes used in this study were NHL5 supplied by St Astier Lime Group, and NHL3.5 and NHL5-Z from Singleton Birch Group Ltd. NHL5-Z is a special product composed of NHL 3.5 or 5 and may contain added pozzolanic or hydraulic materials up to 20% by mass (BS EN 459-1: 2010). The NHL5-Z used in this study is a blend of NHL3.5 and 10% by mass of GGBS, as reported by the manufacturer. Since the density of GGBS is 0.989 g/cm³, % addition by mass is equivalent to % addition by volume and in the rest of this paper will simply be referred to as % addition. All the hydraulic limes examined conformed to the requirements of BS EN 459-1 (BSI, 2010). The GGBS used was supplied by Civil and Marine Ltd, and conforms to BS EN 15187-1 (BSI, 2008). The Portland cement was CEM I manufactured by Hanson cement Ltd and conforms to BS EN 197-1 (BSI, 2000). Sieved mortar sand supplied by Carver Ltd was used, which conforms to BS EN 13139 (BSI, 2002). After drying at 105 °C and sieving, the 300-600 µm size fraction of sand was used to ensure consistency for all mixes. All the measurements of desorptivity were carried out on freshly mixed mortars of composition 0.78: 1: 2 (water: binder: sand) by volume as recommended by the manufacturer to give optimum workability. Replacement amounts of 10, 20, 30, 50, 75 and 100% GGBS were also investigated for an NHL5 mortar.

2.2 Mortar preparation

The densities of the binder and sand materials were determined by weighing different volumes of each material in measuring cylinders (50, 100, 250, and 500 cm³) filled with un-tamped material. The mean density was calculated from four values. The mass of both binder and sand required to produce the mix proportions by volume were then calculated. To ensure consistency, a constant mixing procedure was followed as detailed in Ince (2009).

2.3 Desorptivity measurement

The desorptivity, R, of the freshly-mixed mortars was measured using an American Petroleum Institute (API) pressure cell (API,1998). A full description of the pressure cell method and the experimental details of the measurement of desorptivity are given in Green et al. (1999). A schematic diagram of the API pressure cell and a photograph of the laboratory setup are shown in Figure 1.

![Figure 1](image)

Figure 1. (a) Schematic diagram of the API pressure cell and (b) the laboratory set-up.

The principle of desorptivity measurement is to expel mix water from a freshly mixed mortar contained in a thick-walled stainless steel cylinder by the action of pressurised nitrogen gas. The desorbed water is collected in a container on a top-
loading balance connected to a computer using software (Sarto Connect) which enables the collection of data at constant time intervals set by the user.

The desorptivity, \( R \), at any fixed gas pressure is determined from the slope of the linear part of a plot of the cumulative desorbed volume of water per unit cross sectional area of the cell, \( i \), versus \( t^{1/2} \). Four measurement pressures 0.05, 0.1, 0.15 and 0.2 MPa were used.

2.4 Particle size measurement

Particle size was measured using a Malvern Mastersizer Micro particle size analyzer which uses laser diffraction. The analysis is based on the principle that all particles scatter light at a range of angles which is a characteristic of their size. The instrument calculates the particle size distribution of a powder. The particle size distribution of each binder was measured after dispersing the powder in propan-2-ol, with no surfactants, followed by 10 s in an ultrasonic bath.

3. RESULTS AND DISCUSSION

3.1 Effect of GGBS on desorptivity

Figure 2 shows the variation of the cumulative desorbed volume of water per unit area with the square root of time at 4 applied gas pressures for the NHL5 mortar and is typical of all the materials examined. Figure 2 also clearly shows the gas breakthrough as sudden departure from linearity. It can be seen that the higher the applied pressure, the shorter the time for gas breakthrough to occur.

Figure 3 shows the variation in desorptivity with applied gas pressure for CEM I, NHL5, NHL5-Z mortar and mortar made using GGBS alone as a binder. It can be seen from Figure 3 that the GGBS has the highest desorptivity at all pressures. The desorptivities of the Singleton Birch NHL5-Z mortar (a blend of NHL3.5 and 10% by mass of GGBS) and CEM I are almost identical, but lower than GGBS. The St Astier NHL5 mortar has the lowest desorptivity and is about 40% less than NHL5-Z and CEM I. The desorptivity of NHL5 mortar is about half that of the mortar made using GGBS alone as the binder. It is also shown that the desorptivity increases with an increase in applied gas pressure for all NHL mortars as found previously (Ince et al., 2011).

![Graph showing desorptivity vs pressure for different mortars](image-url)

Figure 3. Variation in desorptivity with applied pressure for hydraulic lime, cement and GGBS mortars.

The NHL5-Z used in this study (as supplied by the manufacturer) is a blend of NHL3.5 and 10% by mass of GGBS, (equivalent to 10 % by volume). This composition can be reproduced in the laboratory by mixing 10% GGBS with NHL3.5. Figure 4 compares the desorptivities of NHL3.5, NHL5-Z and NHL3.5 + 10% GGBS. It can be seen that the desorptivities of the NHL5-Z and NHL3.5 + 10% GGBS are almost identical and that both are only marginally higher than the mortar made with NHL3.5 alone.

![Graph showing desorptivity values for NHL5-Z, NHL3.5 and replicate NHL5-Z (NHL3.5 + 10% GGBS) at four applied gas pressures](image-url)

Figure 4. Desorptivity values of NHL5-Z, NHL3.5 and replicate NHL5-Z (NHL3.5 + 10% GGBS) at four applied gas pressures.

The results in Figure 4 demonstrate the effect of GGBS addition on the desorptivity of NHL3.5 and are further supported by the data of figure 5 which show that the addition of only 10% GGBS has negligible effect on the desorptivity.

Figure 5 shows the variation in desorptivity of NHL5 mortar with varying % additions of GGBS at the same four applied gas pressures. There is no clear effect of GGBS addition on the desorptivity up to 50%, but there is a steady increase in desorptivity with higher percentage additions. The results are essentially the same for each pressure.

![Graph showing desorptivity of NHL5 mortar with varying GGBS addition](image-url)

Figure 5. Desorptivity of NHL5 mortar with varying % additions of GGBS at the same four applied gas pressures.
Appendix B: Conferences papers

3.2 Effect of particle size on desorptivity

Figure 6 shows the particle size distributions of NHL3.5, NHL5-Z, GGBS, CEM I and NHL5. It can be seen that the materials fall into two broad groups: (a) NHL3.5 and (b) NHL5-Z having narrow particle size distributions; and (c) GGBS, (d) CEM I and (e) NHL5 showing much broader distributions.

The finest materials are NHL3.5 and NHL5-Z, with mean particle sizes of 4.32 and 7.11 μm respectively. CEM I, GGBS and NHL5 are the coarser with a mean particle size of 18.11, 15.33 and 15.75 μm respectively. If we compare the desorptivity of the finer particle-size NHL5-Z (which is a commercial mix of NHL3.5 and 10% GGBS) with NHL3.5 it can be seen that the NHL5-Z mortar has higher desorptivity than that of NHL3.5 mortar (as shown in Fig. 4) and the reason may be attributable to the larger particle size of the GGBS component in the NHL5-Z. However, comparing NHL5 and NHL5-Z in Figure 6, it can be seen that the finer NHL5-Z has a higher desorptivity than the coarser NHL5 (Fig. 3). In this case the hydraulicity must be the controlling factor. In the case of CEM I and NHL5, since the particle size is nearly the same for each, it must also be the hydraulicity which accounts for the difference in desorptivity.

The higher desorptivity of GGBS mortar is most likely due to the particle size factor. The particle size distribution of GGBS (c) is quite broad. When mixed with NHL3.5, the particle size distribution of the resulting replicate NHL5-Z becomes broader than that of NHL3.5 alone. This shows that the particle size distribution of NHL3.5 is strongly modified by the addition of GGBS.

The particle size distributions of NHL5, NHL5 + 50% GGBS and NHL5 + 75% GGBS are compared in Figure 7(a). This figure shows that the particle size distribution of NHL5 is strongly affected by the addition of the higher percentages 50 and 75% of GGBS. The particle size distributions of the “replicate” NHL5-Z (NHL3.5 + 10% GGBS) and the commercial NHL5-Z are compared with NHL3.5 in Figure 7(b).

Figure 7 (b) shows the similarity in particle size distribution of the commercial NHL5-Z and replicate NHL5-Z (NHL3.5 + 10% GGBS). Since both NHL5-Zs have the same desorptivity, this confirms that the NHL5-Z can be replicated in the laboratory.

4. CONCLUSIONS

It is possible to examine the effect of addition materials on the water retaining behaviour of
freshly mixed hydraulic mortars in a quantitative way by measuring the desorptivity. The pozzolanic addition GGBS, which is widely used in concretes, makes hydraulic lime mortars less water retaining. When mixed with NHL5 as 10% of binder volume it increases the desorptivity of the mortar by only a small amount. However, increasing the percent addition to 50% increases the desorptivity by nearly 25%. GGBS additions to NHL up to 30% have little discernable effect in terms of the water retaining ability of freshly mixed hydraulic and cementitious mortars.

It has been possible to confirm that the composition of an NHL5-Z may be replicated in the laboratory by combining the separate constituents NHL3.5 and GGBS and comparing the desorptivity and the particle size distributions with those of NHL5-Z mortar of the same mix proportions.

It has been shown previously that coarser sand decreases the water retaining ability of mortars and the results of this study indicate that particle size of the binder material may also be a contributing factor to the water retaining ability of mortars generally.

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REFERENCES


Factors affecting the water retaining ability of hydraulic mortars

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ABSTRACT

Desorptivity defines the water retaining ability of a mortar in the freshly mixed wet state. Desorptivity measurements have been carried out in mortars using an API pressure cell. CEM I is a Portland cement of which clinker composes 95-100% of the constituents. CEM II is Portland-limestone cement of which clinker composes 80% of the constituents and limestone powder the remaining 20%.

A comparison of CEM I and CEM II mortars provides a useful model for assessing the change in water retaining ability of a hydraulic mortar with a non-pozzolanic addition, in this case limestone powder. We present experimental results on the desorptivity of CEM II which is essentially CEM I+20% limestone powder. Reasons for the use of limestone powder as an addition are discussed as well as the likely effects of greater quantities of this addition. We further show how simulated CEM I pore water affects the desorptivity of hydraulic lime binders.

1. INTRODUCTION

The water retaining ability of freshly mixed wet mortars has been derived and quantified by a well-defined parameter, the desorptivity R (Hall and Hoff, 2002). The water retaining ability of fresh mortars can be measured using an American Petroleum Institute (API) pressure cell (American Petroleum Institute, 1998). Previous researchers (Carter M. A. et al., 2003; Collier et al., 2007; Green et al., 1999; Carter et al., 2006) have used this pressure cell technique to measure the desorptivity of cement and lime mortars and have shown that desorptivity depends greatly on the hydraulicity of the binder material used in the mortar mix. Hydraulicity, defined as the ability of the material to set under water, is determined by the amount of clay impurity in the original limestone. The hydraulicity of binders, in order of most to least, is Portland cement, hydraulic limes NHL5>NHL3.5>NHL2. As a result hydrated lime has the greatest water retaining ability, hydraulic lime less and Portland cements the least.

It has been shown that lime-based mortars possess high water retentivity in comparison with cement-based mortars, but there is little explanation of the reasons for this. Hendrickx et al. (Hendrickx et al., 2008) found that the desorptivity of hydraulic mortar inversely proportional with the specific surface area of the binder. Ince et al. (Ince et al., 2011) confirmed previous results that desorptivity depends greatly on the hydraulicity of the binder material and also showed that a decrease in sand particle size caused a progressive decrease in desorptivity of mortars in general. Our previous published work investigates for the first time the effect of binder particle size and addition of the pozzolanic ground granulated blast furnace slag (GGBS) on mortar desorptivity (N. K. Al-Defai et al., 2012).

The addition of pozzolanic materials such as fuel ash, silica fume, glass powder and ground granulated slag as a binder replacement to cement or lime mortars results in increased compressive and bond strength (Fragata et al., 2007; Taha and Shriwe, 2001; Chen et al., 2004; Idris et al., 2011; Targan et al., 2003).

The addition of non-pozzolanic materials such as limestone powder to Portland cement improves the workability and reduces the tendency to bleeding. However the strength of limestone cement mortars and concrete lowered due to the less extent of limestone powder participation in the hydration reaction. This reduction in strength compensated by the finer grinding of cement due to easily ground limestone, but the saving of fuel by substituting limestone for some of the clinker is partially affected by the additional energy required for finer grinding. the existing published researches on the use of lime stone in Portland cement emphasis on amounts of 5% or less (Hawkins et al., 2003) although the European standard
EN 197-1 (British Standards Institution BS En197-1:2011, 2011) allows cement to contain limestone in three different quantity levels. CEM I Portland cement may contain up to 5% minor additional constituents, of which limestone is one possible option. CEM II/A-L and CEM II/B-L, both called “Portland limestone cement,” contain 6-20% and 21-35% ground limestone respectively. However no study has been made until the present work on how these non-pozzolanic materials affect the water retaining ability of freshly mixed hydraulic mortars.

Ince (Ince, 2009) found that the solution extracted from freshly mixed Portland cement paste made with a water: cement ratio of 1:1 using API pressure cell at 0.5 MPa, when used as mix water, increases the water releasing ability of CL90 mortar. Even as little as 2% extract solution added to the mix water was enough to decrease the strong water retaining ability of CL90 pastes and mortars. The chemical analysis of a range of solutions showed that Portland cement extract solution had high sulphate and potassium concentrations. It was also found that the desorvities of CL90 mortars made using potassium sulphate (K₂SO₄) solution as mix water were comparable to values obtained using Portland cement extract solution as mix water.

The purpose of this paper is to demonstrate the effects of the addition of the non-pozzolanic material (limestone powder) on the water retaining properties of freshly mixed Portland cement mortars. In this present study also the effect of potassium sulphate solution of the same concentration as from Portland cement paste on NHL5, GGBS and a mix of 50% NHL5+ 50% GGBS mortars was investigated, with the aim of examining the role of sulphate ions in water retentivity.

2. EXPERIMENTAL DETAILS

2.1 Materials and mix design

Two types of Ordinary Portland cement were used. CEM I manufactured by Hanson Heidelberg cement Ltd, for which clinker comprises 95-100% of the constituents and 0-5% are minor additional constituents (limestone is one possible option). The second type of cement was general purpose Portland-limestone cement manufactured by Lafarge cement Ltd and designated CEM II/A-L for which clinker comprises 80-94%. The range of proportions of the secondary constituent at 6-20% and (LL) signifies a source of higher purity limestone with a low content of total organic carbon (TOC). Both Portland cements conform to the requirements of BS EN 197-1 (BSI, 2011). The proportion of limestone in CEM II cement used in this work is 20% (as reported by the manufacturer). The natural hydraulic lime used was NHL5 supplied by St Astier Lime Group. The hydraulic lime used conform to the requirements of BS EN 459-1 (BSI, 2010).

The pozzolanic material used was ground granulated blast furnace slag (GGBS) supplied by Civil and Marine Ltd, and conforms to BS EN 15167-1 (BSI, 2006). Sieved mortar sand supplied by Carver Ltd was used, which conforms to BS EN 13139 (BSI, 2002). After drying at 105 °C and sieving, the 300-600 μm size fraction of sand was used to ensure consistency for all mixes. Table 1 summarises the composition expressed as oxides and physical properties of the cement and natural hydraulic lime binders and the GGBS pozzolanic addition.

<table>
<thead>
<tr>
<th>Material</th>
<th>CEM I</th>
<th>CEM II</th>
<th>NHL5</th>
<th>GGBS</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO (%)</td>
<td>66.23</td>
<td>61.52</td>
<td>59</td>
<td>40</td>
</tr>
<tr>
<td>SiO₂ (%)</td>
<td>18.85</td>
<td>17.79</td>
<td>15</td>
<td>37</td>
</tr>
<tr>
<td>Al₂O₃ (%)</td>
<td>3.61</td>
<td>4.57</td>
<td>1.92</td>
<td>11</td>
</tr>
<tr>
<td>Fe₂O₃ (%)</td>
<td>2.82</td>
<td>2.87</td>
<td>0.57</td>
<td>0.2</td>
</tr>
<tr>
<td>MgO (%)</td>
<td>2.02</td>
<td>1.4</td>
<td>1.01</td>
<td>16</td>
</tr>
<tr>
<td>SO₃ (%)</td>
<td>3.21</td>
<td>2.87</td>
<td>0.41</td>
<td>-</td>
</tr>
<tr>
<td>K₂O (%)</td>
<td>0.70</td>
<td>0.66</td>
<td>0.21</td>
<td>-</td>
</tr>
<tr>
<td>Na₂O (%)</td>
<td>0.26</td>
<td>0.11</td>
<td>0.07</td>
<td>-</td>
</tr>
<tr>
<td>Ca(OH)₂ (free lime)</td>
<td>-</td>
<td>-</td>
<td>22</td>
<td>-</td>
</tr>
<tr>
<td>CaCO₃ (unburned)</td>
<td>-</td>
<td>17.64</td>
<td>23</td>
<td>-</td>
</tr>
<tr>
<td>Particle size (μm)</td>
<td>0.5-100</td>
<td>-</td>
<td>-</td>
<td>5-30</td>
</tr>
<tr>
<td>Bulk Density (g/cm³)</td>
<td>1.12</td>
<td>1.19</td>
<td>0.75</td>
<td>1.0-1.1</td>
</tr>
<tr>
<td>Specific surface area (m²/kg)</td>
<td>350</td>
<td>361.38</td>
<td>800</td>
<td>400-600</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>1.24</td>
<td>7.61</td>
<td>16</td>
<td>1.0</td>
</tr>
</tbody>
</table>

The high quantity of the unburned limestone in CEM II proves that the addition of limestone powder is 20% as reported by the manufacturer. The quantities of all other minor components are within the limits of the
Appendix B: Conferences papers

2.2 Bulk density measurement

The mass of both binder and sand are required for the mix proportions by volume. Therefore densities of the binders and the sand were carefully determined. The densities were determined by weighing different volumes of each material in measuring cylinders (50, 100, 250, and 500 cm³) filled with un-tamped material. The mean density was calculated from four values. Table 2 compares the measured densities with the densities given by the manufacturer. It can be seen that the quoted densities of all the binders and sand were higher than the measured ones by a small percent (not more than 10%). These differences may be due to the way of handling the material. The bulk density values vary if there is any tamping, vibration or shaking of the container which may settle the particles and give higher mass and as a result higher bulk density.

Table 2. Measured and manufacturers’ quoted values of the bulk density of the binders and sand. Quoted values are from manufacturers (Hanson Heidelberg and Lafarge cement, St Astier lime and Carver sand Ltd).

<table>
<thead>
<tr>
<th>Material</th>
<th>Density values obtained from different volumes of material (g/cm³)</th>
<th>Mean density g/cm³</th>
<th>Quoted density g/cm³</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>50 cm³</td>
<td>100 cm³</td>
<td>250 cm³</td>
</tr>
<tr>
<td>CEM I</td>
<td>0.90</td>
<td>0.94</td>
<td>0.95</td>
</tr>
<tr>
<td>CEM II/A-LL</td>
<td>1.10</td>
<td>1.04</td>
<td>1.13</td>
</tr>
<tr>
<td>NHL5</td>
<td>0.65</td>
<td>0.69</td>
<td>0.67</td>
</tr>
<tr>
<td>GGBS</td>
<td>0.98</td>
<td>0.98</td>
<td>0.98</td>
</tr>
<tr>
<td>Sand</td>
<td>1.39</td>
<td>1.42</td>
<td>1.40</td>
</tr>
</tbody>
</table>

2.3 Mortar preparation

To ensure consistency, a constant mixing procedure was followed as detailed in (Ince, 2009).

Briefly the mixing regime is to add the required volume of water to the bowl first, then the binder material and mixed at low speed for 1 minute. For consistency across all mixes, the mix components were added to the mixer sequentially. Next the sand was added and mixing continued for a further two minutes. Mixing was then continued for a further 7 minutes giving a total mixing time of 10 minutes. The mass of each batch was between 1.2 - 1.6 kg which was sufficient to provide 3-4 sequential desorptivity measurements.

2.4 Desorptivity measurement

The desorptivity, \( R \), of the freshly-mixed mortars was measured using an American Petroleum Institute (API) pressure cell (API, 1998). A full description of the pressure cell method and the experimental details of the measurement of desorptivity are given in (Green et al., 1999). The principle of desorptivity measurement is to expel mix water from a freshly mixed mortar contained in a thick-walled stainless steel cylinder by the action of pressurised nitrogen gas. The desorbed water is collected in a container on a top-loading balance connected to a computer using software (Sarto Connect) which enables the collection of data at constant time intervals set by the user.

The desorptivity, \( R \), at any fixed gas pressure is determined from the slope of the linear part of a plot of the cumulative desorbed volume of water per unit cross sectional area of the cell, \( i \), versus \( t^1/2 \). Four measurement pressures 0.05, 0.1, 0.15 and 0.2 MPa were used.

2.5 Sulphate solution preparation

For the investigation of the effect of mix water chemistry on the water retentivity of NHL5 and GGBS mortars, potassium sulphate solution was prepared and used as mix water. A solution of the same sulphate ion concentration (5.38 g/l) as found in CEM1 extract solution (Ince, 2009) was prepared by dissolving 9.75 g of potassium sulphate in 1 litre of deionised water in a volumetric flask using laboratory grade potassium sulphate, which was weighed out on a two decimal place top loading balance.

3. RESULTS AND DISCUSSION

3.1 Effect of Limestone addition on desorptivity

To show the effect of non-pozzolanic addition on the desorptivity of hydraulic cement mortar, variation in desorptivity with applied pressure was monitored for two commercial types of Portland cement. One type without addition, Ordinary Portland cement CEM I, and another with limestone addition, Portland limestone cement CEM II, which already contains a controlled level of high-purity limestone powder at 20% (manufacturer data sheet). Limestone is at the present time being used in cements produced all over the world. The production of limestone cement leads to reduced levels of CO₂ emissions and reduced energy consumption when compared with CEM I production (Hawkins et al., 2003). The main properties of cements that contain additional constituents are influenced by both the particle size distributions and the chemical/mineralogical compositions of the component materials. Limestone acts primarily as an inert
additive and often does not participate in the hydration reactions therefore the loss in strength of limestone cement is compensated by finer grinding of the cement. The easier grindability of limestone allows a finer particle size distribution of CEM II and this leads to improved workability and water retentivity of the cement (Hawkins et al., 2003).

Figure 1 shows that the limestone powder addition reduces the desorptivity of Portland cement or in other words improves its water retentivity by about 20%. This reduction in desorptivity gets higher at greater pressure (about 25% at 0.2 MPa).

Limestone powder is a non-pozzolanic and non-hydraulic material, and therefore it does not have any effects on the chemical composition or the hydraulicity of cement. CEM II consists of 80% CEMI and 20% of limestone (CaCO₃) which acts as a filler material and therefore the hydraulicity of CEM II is less comparing to CEM I.

![Graph showing desorptivity with applied pressure for two types of cement: CEM I and CEM II (limestone cement).](image)

The specific surface area of CEM II is a little higher (table 1) due to a well-ground limestone powder and this could have an effect on desorptivity (reduces it) according to (Hendrickx et al., 2008).

3.2 Effect of mix water chemistry on desorptivity

In this section the effect of simulated CEM I pore water, used as mix water, on desorptivity is examined.

Green et al. (1999) found that the addition of a small amount of lime to lime greatly increases the desorptivity of lime mortar. They found that the replacement of 30% of lime binder by cement had almost doubled the desorptivity of the 1:3 (lime: sand) mix at 0.5 MPa. Ince, (2009) found that even 2% of Portland cement extract solution added to the mix water was enough to decrease the strong water retaining ability of CL90 pastes and mortars. The study analysed a range of binder materials extract solutions and found that potassium and sulphur (as sulphate) are present in high concentration in Portland cement, less in hydraulic limes and exist only as impurities in the hydrated lime CL90. It was found that the desorptivity values of CL90 mortars made using potassium sulphate (K₂SO₄) solution as mix water were very similar to those obtained by using CEM I extract solution as mix water.

In this work a sulphate solution was used as the mix water to simulate the effect on desorptivity of adding CEM I to hydraulic lime and GGBS mortars. Potassium sulphate solution was prepared of the same concentration as CEM I extract by dissolving 9.75 g of potassium sulphate in 1 litre of water. Table 3 compares the desorptivity of NHL5, GGBS and a mix of 50% NHL5 + 50% GGBS mortars made with either water or K₂SO₄ solution as mix water over a range of applied gas pressures. It is very clear that K₂SO₄ solution increases the desorptivity not only for NHL5 mortar (columns 2 and 3) but also for the GGBS mortar (columns 6 and 7) at all pressures by an amount that depends on the gas pressure used.

<table>
<thead>
<tr>
<th>Pressure (MPa)</th>
<th>NHL5/ water</th>
<th>NHL5/ K₂SO₄ solution</th>
<th>NHL5 + 50% GGBS/ water</th>
<th>NHL5 + 50% GGBS/ K₂SO₄ solution</th>
<th>GGBS/ water</th>
<th>GGBS/ K₂SO₄ solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.05</td>
<td>2.80</td>
<td>3.52 (10%↑)</td>
<td>3.61</td>
<td>3.99 (10%↑)</td>
<td>5.41</td>
<td>5.73 (10%↑)</td>
</tr>
<tr>
<td>0.15</td>
<td>4.33</td>
<td>4.25 (10%↑)</td>
<td>4.71</td>
<td>5.98 (17%↑)</td>
<td>6.30</td>
<td>6.64 (10%↑)</td>
</tr>
<tr>
<td>0.20</td>
<td>5.64</td>
<td>6.02 (10%↑)</td>
<td>6.05</td>
<td>6.84 (17%↑)</td>
<td>7.08</td>
<td>7.60 (10%↑)</td>
</tr>
</tbody>
</table>

![Table showing desorptivity of different mortars compared to water.](image)

Figure 2 presents the NHL5 and GGBS mortar results graphically which makes it clear that the effect of sulphate ions is of similar magnitude on each mortar. These results suggest that sulphate ions are the main cause that is responsible for increasing desorptivity. The higher concentration of sulphate in Portland cement and hydraulic limes is consistent with the fact that the desorptivity of these binders are higher than that of hydrated lime. The mechanisms of this are not yet fully understood. It is likely that the strong (–ve) charge causes an electrostatic effect as seen in the dispersion of clays, silts and others fine particles (river estuaries).
Appendix B: Conferences papers

Figure 2: Variation in desorptivity with applied pressure for NHL5 and GGBS mortars prepared with water or with K\textsubscript{2}SO\textsubscript{4} solution as the mix water.

4. CONCLUSIONS

- It is possible to examine the effect of addition materials on the water retaining behaviour of freshly mixed hydraulic mortars in a quantitative way by measuring the desorptivity.
- The limestone powder addition is an inert filler which slightly reduces the desorptivity of Portland cement.
- Adding sulphate ions to the mix water of hydraulic lime mortars reduces their water retaining ability, as has been previously found for hydrated lime mortars. Sulphate also reduces $R$ of mortars made with GGBS as binder. It may be suggested that the strong negative charge of sulphate causes an electrostatic effect.

REFERENCES


