Enhancing the understanding of lime stabilisation processes

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Enhancing the understanding of lime stabilisation processes

Paul Beetham
ENHANCING THE UNDERSTANDING OF LIME STABILISATION PROCESSES

By
Paul Beetham

A dissertation thesis submitted in partial fulfilment of the requirements for the award of the degree Doctor of Engineering (EngD), at Loughborough University

August 2015

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Lastly, thanks to the Britpave soil stabilisation task group for allowing me to join your meetings and share my findings with the wider industry.

Dedicated to Nana.
Enhancing the understanding of lime stabilisation processes

ABSTRACT

Lime stabilisation is a ground improvement technique used to improve the engineering properties of cohesive fill material. During earthworks operations, specialist plant is used to rotovate the material and intermix lime binder around the resulting clay clods. After completion of the lime treatment, the layer is compacted in the usual way. Immediately after mixing, the lime instigates a series of physico-chemical reactions within the clay soil. Where the chemical reactions are favourable and with time after compaction (curing) the material becomes progressively stronger and durable to environmental influences, e.g. inundation by surface or ground water. However, where sulphate is present within the soil, the reactions may change and the ingress of water into the layer can result in the expansive growth of deleterious minerals e.g. ettringite.

While sulphate swell issues are relatively rare, when they do occur the degree of expansion can be very high. A high profile sulphate swell failure developed during the construction of the M40, Oxford, UK in 1989. Over the winter period after the lime stabilisation works, a 250mm deep lime treated layer heaved by up to 150mm - destroying the overlying road construction. Since the M40 failure, a substantial amount of effort has been undertaken to better understand the sulphate swell reactions and in this regard the state of scientific knowledge is relatively strong.

A fundamental issue for field applications of lime stabilisation is that the vast majority of research has been undertaken on laboratory specimens prepared using methods which do not reflect site practice. Laboratory studies often use oven dried and finely crushed clay, whereas site operations will treat much larger clay clods to result in a more heterogeneous distribution of lime through the compacted soil body. With large clay clods, the chemical reactants must migrate through clods and this may cause the sequence of chemical reactions to change. A further challenge is that laboratory studies are typically undertaken with cure temperatures of 20°C, whereas a typical near surface temperature in the UK is <10°C. This is of particular relevance to sulphate swell failures which are reported to coincide with a reduction in ambient temperature over winter periods. Thus, the direct relevance of laboratory studies to site application was unclear.

A series of laboratory experiments using a preparation method which reflects field applications of lime stabilisation was used to investigate the influence of large clay clods on the durability of lime stabilised clay soil. This method was applied to both low and high sulphate clay soils.

A fundamental discovery from work on low sulphate clay is that the addition of lime binder to the surface of the clay clods causes a physico-chemical boundary to form. This boundary develops due to the rapid increase to the plastic limit of the clay preventing adjacent clods from joining together during compaction. This causes the engineering properties of each individual clod to develop independent to its neighbours and for each clay clod to be separated by an inter-clod pore space. The strength of each individual clay clod will increase with curing as the added lime dissociates into Ca^{2+} and OH⁻ and migrates to form C-S-H deep within the
clods. Where the material is compacted wet of the optimum water content, this condition improves ion migration and enables development of diffuse cementation deep within clods. The inter-clod porosity remains as a weakness throughout curing especially during specimen soaking, where the pore channels comprise a pathway, accelerating the ingress of soaking water. With low sulphate soil, the soaking water softens the treated material, however, with high TPS soil substantial sulphate swelling may develop. Thus, efforts to minimise this porosity during preparation is important and the use of quicklime with longer mellowing periods can cause the clay clods to develop high strength before compaction. The high strength clods resist compaction and the degree of inter-clod porosity in the compacted mass increases, worsening specimen durability to water ingress.

The investigations into high sulphate clays included the development of a ‘Novel Swell Test’ (NST) to assess volume change. A unique aspect of the NST was that the sulphate swell response of the lime treated material was investigated at site realistic temperatures of 8°C. It was identified that, when compared with standard laboratory test temperatures of 20°C the rate of sulphate swell is substantially higher at the low temperature. The mineralogical testing has permitted the hypothesis that, at 8°C the growth of crystalline ettringite becomes slower and the ettringite precursor, which has a high affinity to imbibe water, remains in this state for much longer. Thus, laboratory swell tests at 20°C may substantially underestimate the degree of swell that may develop in the field. As a pressing need, it is recommended that the industry adapt sulphate swell test methods to appraise the degree of swell at field realistic temperatures i.e. <10°C. The work also identifies that the primary defence against sulphate swell is to condition the fill so that the risk of post compaction water ingress, via inter-clod porosity, is minimised. The use of GGBS and water addition during extended mellowing periods also reduces the degree of sulphate swell in natural clay soils.

This work concludes that working methods for lime stabilisation of medium – high plasticity soils of a potentially high sulphate content, should be adapted to encourage diffuse cementation and minimise the degree of (post compaction) inter-clod porosity. Practically this involves the use of hydrated lime and the addition of mixing water throughout extended mellowing periods. Fundamentally, the study recommends that where construction programmes allow, the long term durability of a fill material should be the priority over immediate strength.

KEYWORDS

Lime stabilisation, ettringite, diffusion, durability, ground improvement, sulphate swell
PREFACE

This thesis culminates research undertaken between 2010 and 2014 in partial fulfilment of the requirements for the award of the degree Doctor of Engineering (EngD), at Loughborough University, Leicester, UK.

The research was funded by the Engineering and Physical Sciences Research Council (EPSRC) through the Centre of Innovative and Collaborative Engineering (CICE), The Independent Stabilisation Company Ltd and Opus International Consultants Ltd.

The EngD award requires that at least three academic papers (including one journal paper) are published over the study period. The thesis presents these papers within the appendices and the thesis text is used to explain the overall research programme, present any unpublished work, and link all work together, including key points from the publications, into a discourse. Finally, the thesis will demonstrate the impact the research has made upon the relevant industry.

This thesis describes the research arising from industry working and two substantial laboratory investigations into the physical and chemical reactions arising from lime stabilisation of clay soils. This research placed a particular emphasis on the desire for industry practitioners to better understand and avoid the deleterious behaviour which may result from the lime stabilisation of clay soils of high sulphate content.
# USED ACRONYMS / ABBREVIATIONS

<table>
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<th>Acronym</th>
<th>Meaning</th>
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<tr>
<td>AST</td>
<td>Accelerated Swell Test</td>
</tr>
<tr>
<td>Ay</td>
<td>Aylesbury Clay</td>
</tr>
<tr>
<td>CBR</td>
<td>California Bearing Ratio</td>
</tr>
<tr>
<td>CG</td>
<td>Caolndon clay reconstituted with 5% gypsum powder</td>
</tr>
<tr>
<td>C-A-S-H</td>
<td>Calcium Aluminate Silicate Hydrate</td>
</tr>
<tr>
<td>C-S-H</td>
<td>Calcium Silicate Hydrate</td>
</tr>
<tr>
<td>GGBS</td>
<td>Ground Granulated Blast Furnace Slag</td>
</tr>
<tr>
<td>HA</td>
<td>Highways Agency (UK)</td>
</tr>
<tr>
<td>ICP-AES</td>
<td>Inductively Coupled Plasma with Atomic Emission Spectroscopy</td>
</tr>
<tr>
<td>IP</td>
<td>Plasticity index (i.e. the percentage difference between the W_L and W_P)</td>
</tr>
<tr>
<td>ISC</td>
<td>Independent Stabilisation Company</td>
</tr>
<tr>
<td>MCDHW</td>
<td>Manual of Contract Documents for Highway Works</td>
</tr>
<tr>
<td>MDD</td>
<td>Maximum Dry Density</td>
</tr>
<tr>
<td>MCV</td>
<td>Moisture Condition Value</td>
</tr>
<tr>
<td>NST</td>
<td>Novel Swell Test</td>
</tr>
<tr>
<td>OIC</td>
<td>Opus International Consultants</td>
</tr>
<tr>
<td>OWC</td>
<td>Optimum Water Content</td>
</tr>
<tr>
<td>PFA</td>
<td>Pulverised Fuel Ash</td>
</tr>
<tr>
<td>SEM-EDX</td>
<td>Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy</td>
</tr>
<tr>
<td>SLS</td>
<td>Surface lime stabilisation</td>
</tr>
<tr>
<td>TGA</td>
<td>Thermogravimetric Analysis</td>
</tr>
<tr>
<td>TPS</td>
<td>Total Potential Sulphate</td>
</tr>
<tr>
<td>W_L</td>
<td>Percentage water content at the liquid limit</td>
</tr>
<tr>
<td>W_P</td>
<td>Percentage water content at the plastic limit</td>
</tr>
<tr>
<td>XRD</td>
<td>X-Ray Diffraction</td>
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LIST OF PAPERS

The following papers, included in the appendices, have been produced in partial fulfilment of the award requirements of the Engineering Doctorate during the course of the research.

PAPER 1 – JOURNAL PAPER (Appendix A)


PAPER 2 - CONFERENCE PAPER (Appendix B)


PAPER 3 - CONFERENCE PAPER (Appendix C)

1 BACKGROUND TO THE RESEARCH

This chapter provides a general introduction into the technique of lime stabilisation and how this process is applied in general accordance with the UK Highways specification by specialist contractors in the UK. While lime stabilisation usually results in the improvement sought, some failures have occurred when high clay soils of high sulphate content have been treated. The context of the failures and a need to better understand how beneficial and deleterious processes develop under field conditions of lime stabilisation is then set. The reader is also given guidance on how the thesis is structured including how the appended publications are linked with the main thesis text.

1.1 THE GENERAL SUBJECT DOMAIN

Lime stabilisation is a form of soil stabilisation, a ground improvement technique that may be used in civil engineering projects. Soil stabilisation involves the inter-mixing of a chemical binder within a body of soil with the intent to improve the engineering performance of the treated material. With lime stabilisation, the lime reacts with clay minerals to instigate a series of physico-chemical processes which improve a clay-soils engineering properties, e.g. improved strength, stiffness and a reduced potential for shrink/swell (Bell 1988). Some of this improvement is almost instantaneous and a low strength, wet clay fill may be transformed into a high strength material within minutes (strength terms as defined in (BSI, 2010). For most clay soils, chemical reactions between lime and clay minerals will continue for months and years, leading to ongoing, gradual improvements in strength. The processes responsible for the rapid change in properties are different to those that cause the long term strength increase. However, both processes have the potential to substantially influence the durability of a treated body, as will be extensively discussed throughout the main text of this discourse.
While lime will typically react well with clay minerals, it is largely ineffective on the granular components of a soil (i.e. sands / gravels; Bell 1988). Where soils are predominantly granular, usually stabilisation is instead undertaken with Portland cement as the binder. Thus, cement stabilisation of granular fill is analogous to a naturally cemented sandstone, and the strength of the soil body increases due to these inter-granular cement bonds. Where natural soils comprise an intermediate mixture of clay minerals and granular constituents (e.g. a clay soil of low plasticity) soil stabilisation may be effective with either lime, cement or lime and cement stabilisation. Furthermore, lime (Ca[OH]₂) is produced during the hydration of Portland cement, so where cement stabilisation is applied to a clay soil of low plasticity, lime-clay reactions will be an unavoidable by-product. Thereby, while the focus of this research is on treatments where lime is used as the primary binder, some of the findings will likely be relevant to other varieties of soil stabilisation e.g. cement stabilisation.

Lime stabilisation may be applied at either the ground surface or at depths below and it is the former that this research is focused on. Deep soil mixing or lime columns, involve the application of the treatment below the ground surface i.e., without recovery of the treated material to surface. With surface techniques the binder is usually mixed into relatively shallow (e.g. 300mm) layers of soil. As the binder addition/mixing stage of surface lime stabilisation (SLS) is applied at an unconfined surface, the subsequent compaction of the treated layer to reduce air voids is an important element of the technique. Thereby, with regard to the field behaviour of materials produced using SLS techniques, it is possible for either compaction, or the lime-clay reactions to influence engineering performance. As a key focus, this EngD will detail the chemical reactions between a lime-clay soil system and then discuss how site working methods, including compaction, may be managed in order to optimise the long term engineering properties of a compacted mass.
1.2 LIME IMPROVEMENT AND LIME STABILISATION

The inter-mixing of quicklime powder with clay soil instigates three separate, but linked processes which causes enhancement of the engineering properties of the treated material. A basic description of each is used to assist subsequent explanation of the SLS site process (see Section 1.3). Table 1.1 summarises the process effect, timescale and an indication of the lime quantity required: (Further detail on these processes are provided in Section 4.1.1 and Paper 1; section 2 adds further site context)

1.2.1 DRYING.

The reaction of quicklime with water is an exothermic reaction and the heat generated rapidly conditions (within minutes and hours) a wet fill material to the optimum water content (OWC) for compaction.

1.2.2 CATION EXCHANGE / MODIFICATION.

The calcium sourced from the added lime causes chemical alterations at the surface of clay particles. As a result, the clay mineral structure alters and the volume change potential of the clay minerals reduces along with reductions to plasticity. This process is relatively rapid to commence, although depending on the clay mineralogy type and lime binder used, may take several days to substantially complete.

1.2.3 Pozzolanic Reactions

When the lime added to the clay soil is greater than the initial consumption of lime value (ICL), pozzolanic reactions, which is the formation of cementitious compounds, may commence. The ICL value (as per the method described BSI, 1990b) corresponds to the amount of lime consumed by any initial reactions with the clay soil (e.g. organic matter may react with OH-
anions), and then elevate the pH of the clay-soil pore water to 12.4 (Eades and Grim, 1966). The highly alkaline environment causes calcium (from the added lime) to react with aluminosilicates within the clay minerals, forming cementitious compounds that bind clay aggregations together. These reactions are relatively slow to progress, but when sustained improve the engineering properties of the fill material for months and years after compaction.

<table>
<thead>
<tr>
<th>Treatment Intent</th>
<th>Physico-chemical Process</th>
<th>Common terminology</th>
<th>Indicative lime requirements</th>
<th>Typical time required*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lower the water content of a wet / low strength soil towards OWC. Either for compaction as a general fill, or to enhance trafficability.</td>
<td>1. Removal of free water by reaction with quicklime; 2. Cation exchange / clay mineral aggregation effectively increasing the OWC.</td>
<td>Lime Improvement</td>
<td>Low, e.g. 0.5-4%. (initial water content / clay content dependent)</td>
<td>1. Immediate 2. Rapid (0-72 hours)</td>
</tr>
<tr>
<td>Reduced plasticity / potential for volume change.</td>
<td>3. Cation exchange / clay aggregation reduces clay mineral effective surface area and affinity for water. 4. Early pozzolanic reactions restrict subsequent dispersion of aggregations</td>
<td>Lime Improvement</td>
<td></td>
<td>Rapid (0 – 72 hours)</td>
</tr>
<tr>
<td>Substantially improve engineering properties e.g. high strength and durability.</td>
<td>5. Pozzolanic reaction within lime-clay soil system.</td>
<td>Lime Stabilisation</td>
<td>In excess of the ICL value, e.g. 2 – 10%; (actual binder addition determined by site specific mix design)</td>
<td>On-going improvement from 72 hours continuing for months / years</td>
</tr>
</tbody>
</table>

* Indicative times only. Actual time depends upon multiple factors detailed in later text.

Table 1.1 Lime treatment intent, implications for lime addition and time dependent reactions.

### 1.2.4 TERMINOLOGY

The rapid improvements to the soil’s engineering properties from drying and cation exchange processes are commonly referred to as “lime improvement” (Table 1.1). “Lime stabilisation” distinguishes that the treatment will add sufficient lime to permit the pozzolanic reactions to develop and is a superior treatment.
1.3 SITE PROCESS

This section first describes the general site approach to SLS. Thereafter, sub headings are used to explain some further stages which are relatively common, but may not be necessary for all treatments.

1.3.1 GENERAL APPROACH

The physical process of SLS can be considered an extension of the methodology utilised in standard earthworks. During a standard earthworks operation, layers of suitable fill material are mechanically compacted to achieve an increased density and improve engineering properties. Achievement of which with standard compaction plant requires that fill layers are relatively thin i.e. 100mm to 300mm (Highways Agency, 2009). SLS adds three additional stages (Sections 1.3.1.1-1.3.1.3, prior to compaction, where the 300mm deep layer is processed by specialist mixing plant moving through the fill at slow walking pace. Historically, these stages were performed by 3 separate pieces of plant (spreader, mixer and water bowser), however, modern plant can deliver all operations within one machine (e.g. Wirtgen WR 2500 SK; Figure 1.1)

1.3.1.1 Binder addition

The binder is spread at the required rate (quantity determined by pre-contract and / or on-site testing; section 1.3.1.4) onto the surface of the treated clay layer, through a feed hopper located ahead of the mixing drum (Figure 1.2).

1.3.1.2 Pulverisation and Mixing

A rotating drum powered by a high torque drive and housing multiple teeth, denudates the full depth of fill, forcing this material upwards and into the mixing drum. The mixing drum has
Enhancing the understanding of lime stabilisation processes

Figure 1.1 A modern soil stabilising machine - Wirtgen WR 2500 SK. The mixing drum (see Figure 1.2) is positioned central to the front/rear wheels and at the base of the machine.

Figure 1.2 Sketch showing a cross section through an integral feed hopper and mixing drum of a Wirtgen WR 2500 SK (reproduced with permission from the WR 2500 SK handbook).
two effects; efficiently mixing the binder with the soil and further pulverising the soil. The term ‘pulverisation’ is often used to describe the breakdown of large lumps into smaller clods.

1.3.1.3 Mixing water addition

Where additional mixing water is required it is applied through water nozzles integral to the mixing drum. The ongoing mixing action allows distribution around the clods.

1.3.1.4 Compaction and Quality Control

When the material has been treated in accordance with the project requirements, and the pre-compaction validation testing is passed (Table 1.2), the material is fully compacted.

For quality control, it is a usual that UK specifications (see section 1.4.2) require pre-contract laboratory testing and the site operation is then correlated to this. The typical scope of pre-contract testing is to confirm the fill is suitable for SLS and then determine the percentage of lime required to achieve the performance requirements. The primary performance requirement is often attainment of a minimum value of laboratory California Bearing Ratio (CBR) after soaked curing (see section 1.4.1 for further information on CBR) although sometimes other strength criteria are specified (e.g. minimum friction angle from shear box tests). Specifications (see section 1.4.2) also usually require that the on-site process conditions the water content of the fill material to a range between 1 to 1.2 times the OWC for compaction. The means of assessing water content on site is usually through correlation with the Moisture Condition Value test (MCV; as described by BSI, 1990c). An MCV of 12-13 will typically correspond with the OWC for most lime treated fill materials, and an MCV of 8 will usually correspond to 1.2 times OWC (Highways Agency, 2007). Thus, the majority of pre-contract laboratory testing regimes will trial a range of batches at different lime binder contents, with sub batches conditioned to water contents across the full MCV8-12 range. A specimen for CBR testing (and any other performance tests) is then prepared from each sub batch.
Table 1.2 Pre-contract suitability and validation tests typically undertaken on SLS contracts

Once CBR values for each sub batch are determined, the required binder content can be selected on the basis that the performance criteria will be satisfied over the full range of water contents that will be achieved by the site operation.

With the baseline provided by the pre-contract lab testing, Pre-compaction verification checks confirm that required binder rate, pulverisation requirements and an MCV value within the prescribed range e.g. 8-12 is achieved. A CBR specimen is also prepared from the treated fill so that a laboratory soaked CBR test can be performed after curing. Post compaction core cutter samples are taken for laboratory determination of dry density / air voids and plate tests verify the in-situ CBR value.

1.3.2 MULTIPLE MIXING STAGES

A contractor would typically seek to pass the validation tests with the minimum number of mixing plant passes as this lessons time, fuel use and wear to the plant. When the intent of the treatment is the lime modification of wet clay to the OWC for compaction, then it is often possible to achieve this with a single pass. However, it may be necessary to mix the treated
material at least twice i.e. with further passes of the rotovating/mixing machinery. Common scenarios requiring multiple passes are:

- When a large amount of binder is required
- Two different binder types are required (e.g. lime and then cement)
- If pulverisation requirements (Table 1.2) are not met with one pass a further pass/passes with or without further quicklime (depending on the MCV) may be used to further pulverise the material.
- To add mixing water if the material has become too dry

1.3.3 MELLOWING

Applications of SLS may include a mellowing phase. “Mellowing is the period of time when a cohesive material and lime mixture is left undisturbed after light compaction” (The Highways Agency, 2007 p. 1/2). The mellowing period commences immediately after the initial inter-mixing of lime and ends after the prescribed duration (often 24 to 72 hours) with a remixing stage. The remixing stage may also include addition of further binders / mixing water before final compaction.

The inclusion of a mellowing period in SLS treatments is sourced from early (1950’s) field observations in the United States that this stage improves pulverisation (Holt & Freer-Hewish, 2000). The Highways Agency (2007) suggest that the mellowing period also allows time for the migration of lime through the clods and for improvement processes to take place and lime migration is considered further in the experimental work of this EngD (See Section 4.2.2).
1.3.4 **OVER-LAPPING TREATMENT BAYS**

Where the intent is to undertake SLS across a large area, the plant dimensions provide a limit to the width of each treatment bay and this would typically be a maximum of 4 metres wide. The plant can extend the treatment bay in a linear direction as far as necessary or until the binder hopper runs empty. So where sites are relatively free of obstacles, the plant can undertake long uninterrupted ‘rips’. When it becomes necessary to treat a virgin area adjacent to a previously treated bay, the usual practice is to ensure all material receives the required binder by overlapping the treatment by at least 0.6 metres. Therefore, this overlap will receive a ‘double dose’ of binder. Where the binder is quicklime, this can cause the overlapped region to become significantly drier than the adjacent material which, following compaction, may result in higher degree of air voids in this zone.

1.4 **SPECIFICATIONS AND QUALITY CONTROL**

SLS in the UK was first used in the 1970’s in the construction of pavement foundation layers (Notman, 2011). More recently, the application of SLS has diversified to include additional purposes, e.g. excavation, stabilisation and recompaction of soft and consolidation susceptible ground beneath residential or commercial structures. However, as the formative use of the technique related to highways construction, much of the literature and specifications relate to pavement foundation layers. It is due to this history that UK specifications for SLS typically include a requirement for a minimum value of California Bearing Ratio (CBR).

1.4.1 **CALIFORNIA BEARING RATIO (CBR)**

A CBR value is determined from a laboratory CBR test (as described by BSI, 2004a), although in situ determination of CBR may also be made. The laboratory method is briefly summarised as follows. The CBR is an index test which relates the results of a tested material to those from
Background to the Research

A standard crushed limestone which is indexed at 100%. Force is applied with a constant strain rate via a 50mm platen and onto the surface of a compacted specimen (confined within a 150mm diameter, 127mm high steel CBR mould). Regular readings are taken in order to produce a force/displacement curve and then back calculate the CBR value from force/displacement curves from the standard crushed limestone.

When CBR tests are undertaken on lime stabilised material, specifications (see Section 1.4.2) usually require that the sample is cured for a set time period, including a phase of full immersion in water (soaking), before testing. This is to allow the time dependent reactions to progress and also to assess the durability of the specimen to saturation. Standard cure periods are 7 days (including 3 days soaking) and 31 days (including 28 days soaking).

There is a long history of using CBR values to determine the depth of pavement foundation layers with empirical correlations (Edwards, 2007). While the relevance of pavement design methods based around determinations of CBR are disputed, the advantages of the test are its low cost, simple execution and high repeatability (Edwards, 2007).

1.4.2 Highways Agency Specification

A major influence upon all SLS work undertaken in the United Kingdom is the Manual of Contact Documents for Highways works (MCDHW; The Highways Agency, 2009). The MCDHW is a comprehensive series of specifications which outline how works shall be executed in order to meet the HA’s requirements. Of specific relevance to SLS is the MCDHW section on earthworks (series 600) and guidance note HA74 “Treatment of fill and capping materials using lime or cement of both” (The Highways Agency, 2007)

Series 600 (The Highways Agency, 2009) includes a ‘method specification’ approach to earthworks. The basis of the method specification is that fill material is first classified with a series of pre-contract tests e.g. particle size distribution, Atterberg tests, water content. This
permits a fill type, e.g. Class 2B, to be assigned to the material, and when the size and type of compaction plant is known, the data tables in series 600 are used to determine the maximum depth of fill layer and the number of passes the compaction plant must make. Where contractors follow the method prescribed, the need for ‘end-product’ testing relating to the attainment of a minimum air voids or maximum dry density (MDD) is typically avoided. This is usually preferred where the specific gravity of the source fill material may vary considerably over the work area (a common scenario in UK earthworks) and validation tests relating to dry density / water content relationships are difficult to enforce. Where a method specification approach is favoured, the lack of any alternative method which commands the trust of contractors and overseeing organisations often leads to the use of series 600 as the baseline contract specification. This is common for earthworks / pavement construction for many civil engineering projects and not just those where the HA is the client. Thereby the MCDHW (The Highways Agency, 2009) is an influential specification, as reflected by references to the document as a ‘de facto standard’ (Bond and Harris, 2008).

For SLS, Series 600 contains clauses specifying how lime treatment may be included within the method specification approach to earthworks. Furthermore, HA74 (The Highways Agency, 2007) sets out further pre-contract tests and performance requirements (Table 1.2 shows some of the required tests). Of specific note are requirements for material used in lime stabilised capping layers to achieve a minimum soaked CBR value of 15% (on average) with no individual test <8%. Aspects of HA74 (The Highways Agency, 2007) relating to sulphate swell are discussed in sections 1.6.1.2

Consequently, a specification (either client, contractor or designer proposed) for SLS works in the UK, whether highways related or otherwise, is likely to be based on the clauses in series 600 and the requirements of HA74.
1.4.3 **OTHER SPECIFICATIONS**

At the time of writing there are two other significant documents under draft.

A specification for lime stabilisation applications on residential housing developments is being prepared by the Building Research Establishment on behalf of the National Home Builders Council. As residential structures may be more sensitive to volume change, i.e. settlement or heave, the resultant specification may include different performance requirements to the CBR value focus of the MCDHW (The Highways Agency, 2009). Although, there is currently no indication of how this may differ, if at all.

Also under draft is a European Standard for Earthworks, which will feature a part on “Soil Treatment with Lime and/or Hydraulic Binders”. To date, the draft content has not been made widely available. However, as the working committee includes representatives from the UK soil stabilisation industry, it is likely that the content will be sufficiently flexible to allow compatibility with current UK practice (focused around the MCDHW; The Highways Agency, 2009) as well as the practices of other European Union member countries.

1.5 **THE INDUSTRIAL SPONSOR**

The project has two industrial sponsors. These are; Consulting Engineers, Opus International Consultants (OIC); Ground Improvement Contractors, The Independent Stabilisation Company (ISC).

Both sponsor companies have strong interests in the science underlying soil stabilisation. Soil stabilisation projects comprise approximately 80% of ISC’s annual turnover, so the desire to improve technical knowledge is core. As multi-disciplinary consultants involved in design, specification and supervision of all aspects of civil engineering, OIC command strong knowledge of earthworks and soil stabilisation.
The needs of both sponsors also require the research to expand beyond scientific development and output in a manner which may impact upon industry guidance. Both Consultants and Contractors must be aware of industry specifications and guidance. Departure from accepted guidance carries a risk of claims and / or lost contract awards. Accordingly, the EngD must be understanding of the constraints that these guidance documents apply. If conflicts between study findings and guidance transpire, it must then become the intent of the EngD to influence these documents, including those under draft. This necessitates that industry peers outside the sponsor companies accept the work.

1.6 THE CONTEXT OF THE RESEARCH

1.6.1 SULPHATE SWELL

While the vast majority of SLS applications are successful and result in attainment of the required engineering performance, a small number of project experiences have resulted in vastly different physical behaviour. High profile failures of SLS works in the UK occurred during the 1989 construction of the M40 Motorway in Oxford and the 2002/3 construction of the A10 Wadesmill bypass.

1.6.1.1 M40

With the M40 motorway, during the winter period following the 1989 lime stabilisation works, a 250mm deep, lime stabilised capping layer heaved by up to 150mm, damaging the overlying road construction which required full reconstruction (Snedker, 1996). A substantial reduction in shear strength was also associated with the damaged areas of capping layer. Remediation of the M40 failure resulted in substantial cost and time delays to the client (the HA; then Department of Transport). To reduce the risk of future works repeating this failure, the HA became involved with studies intending to find ways of adapting their specifications to include
necessary precautions. The causality of the M40 failure was attributed to chemical reactions between the pyrite (iron sulphide) bearing Lower Lias clay soils, the added lime and surface water soaking the treated layer (Snedker, 1996). The product of these reactions was an expansive mineral e.g., ettringite and/or thaumasite, and it is the sustained formation of these minerals which was associated with the substantial heave recorded. Herein this type of failure shall be referred to as sulphate swell or swelling.

1.6.1.2 Changes After The M40

At the time of the M40 failure, the potential for sulphate swell reactions was not reflected in the HA specification and the risk was little recognised by the UK industry. A revision of HA74 in 1995 was used to add detail on the key issues identified by the investigations into the M40 failure. This included:

- Information on how sulphate swelling may develop in lime treated soil;
- Detail on site investigation approaches to identify potentially high sulphate ground, including a focus on the potential for sulphate to form via the oxidation of pyrite (as was the case with the M40);
- Recommendation to use soaked CBR tests as a pass/fail test in the determination of sulphate swell risk.

Furthermore, the publication of report TRL447 “Sulphate specification for structural backfill” (Reid et al, 2001) signified a major improvement to the methods of identifying and testing sulphate levels in soils. One of the major challenges prior to this point had been quantifying the different forms of sulphur within soil. The quantification of the total amount of elemental sulphur within a soil is not necessarily useful, as it is only the sulphur present as sulphate ($SO_4^{2-}$) that can contribute to sulphate swelling reactions. However, where the fill contains sulphur
present as sulphide (as with the pyrite oxidation on the M40), the lime stabilisation process may rapidly oxidise the sulphide to form sulphate. Therefore, it is the total potential sulphate (TPS) % value i.e. the amount of sulphate present from naturally occurring sulphate (e.g. gypsum) in addition to sulphate which may oxidise from sulphides, which is of relevance. TRL447 (Reid et al., 2001) was useful in clearly explaining the challenge of determining sulphur species and also through description of repeatable, rapid and cost effective test methods to accurately determine the TPS % value in soil. The latter was most important as previous test methods were regarded as either unreliable, or slow and expensive which was a real hindrance to determining the potential sulphate swell risk in pre-contract investigations.

As the presence of sulphate within the lime stabilised clay soil was identified as the primary cause of sulphate swell, revision of HA guidance notes also introduced threshold values of TPS for the fill material. Beyond the lower thresholds further testing is required, or at the higher level SLS is not advised. HA44 (“Earthworks design and preparation of contract documents) p10/8 states that the maximum TPS value “should normally be 1.0% except where, case studies or a prolonged trial indicate that a higher value is acceptable” (The Highways Agency, 1995). For the purpose of this thesis, a fill material with a TPS >1% shall herein be referred to as a high TPS material. The lower threshold of sulphate swell risk was set where the TPS value exceeds 0.25% and soaked CBR tests to determine if the degree of sulphate swell risk is acceptable are advised. With the soaked CBR validation, the amount of swell is measured during the soaking phase of the test and if the average from all tests is <5mm, with no individual test result swelling >10mm, the risk is considered acceptable (The Highways Agency, 2007).
1.6.1.3 A10 Wadesmill bypass

The sulphate swell at the Wadesmill bypass followed a similar pattern to the M40. Following on the spring/summer 2003 SLS works and subsequent completion of the overlying carriageway, damage from sulphate swell was first reported in early winter (November 2003; Hawkins, 2013). The heave totalled 80mm and required full reconstruction of the pavement.

As with the M40, the substantial source of the sulphate was from the oxidisation of pyrite. The pre-contract testing (which did not include testing for oxidisable sulphate from pyrite) recorded a maximum sulphate value of 1.2%. However, post failure testing revealed the sulphate content, including oxidised sulphide, to be as high as 4.3% (Pritchard et al., 2013). Thus, in spite of increased industry awareness, changes to the specifications and improvements to sulphate test methods, the risk of sulphate swell was not correctly identified and the failure resulted.

1.6.2 DEVELOPMENTS IN SULPHATE SWELL SUPPRESSION

Since the M40 failure, there has been a significant amount of research reporting that the use of a secondary binders e.g. Ground Granulated Blast Furnace Slag (GGBS) with SLS substantially reduces the degree of sulphate swell in high TPS soils e.g. (Wild et al., 1999; Wild et al., 1998; Higgins, 2005). Laboratory studies have shown that with a lime/GGBS treatment, clay soils with 3% TPS can achieve the soaked CBR swell test criteria stipulated in HA74 (Higgins and Kennedy, 1998). Furthermore, field applications using lime / GGBS on soils with TPS value of up to 2% have been undertaken without reported incidence of sulphate swell (Britpave, 2005).

HA74 (The Highways Agency, 2007) does note that lime/GGBS use may reduce the risk of sulphate swell, however, this specification does not suggest how working methods should be adapted to include GGBS use, nor whether an increase to the 1% TPS threshold is
permissible. Britpave (2005) do advise adaptations to working methods for high TPS soils, and this advice relates to the use of extended mellowing periods (>48 hours), the addition of further mixing water during mellowing and, where GGBS is used, the addition of the GGBS in a second stage after mellowing.

1.6.3 SPONSOR COMPANY EXPERIENCE OF SULPHATE SWELL

Prior to commencement of the EngD research, ISC had noted some disparity between the sulphate thresholds in the HA guidance documents and past working experiences of stabilising high TPS fill. ISC staff were involved with the M40 and other (non-published) failures where sulphate swell had been attributed as the cause. Additionally, prior to the introduction of the sulphate thresholds in HA guidance, ISC staff had experience of lime and / or cement stabilisation projects subsequently known to have a TPS >1%, but where swell problems were not identified. This working knowledge of failed and successful lime treatments of high TPS material allowed ISC to associate some additional factors relating to the development of sulphate swell failure. These are summarised.

1.6.3.1 Environmental influences

As was noted with the M40 failure (Snedker, 1996), the stabilised material initially appears satisfactory both on site and from routine laboratory tests (7 and 31 day soaked CBR tests do not indicate any swell >5mm). However, the on-site heaving will then commence and develop substantially over the winter period after the treatment. Thereby, seasonal change to temperature and / or rainfall may be triggers. Furthermore, the suitability of soaked CBR tests to replicate the site conditions associated with the very large degree of swelling has been questioned (Notman, 2011).
ISC also consider site location and drainage to comprise a key factor. Failure locations are often at low points where surface water may locally or regionally accumulate. ISC advise how the post-failure installation of a site perimeter drainage system successfully remediated a site that was experiencing ongoing sulphate swell. The drainage of surface water through cut slopes and into regions where the sulphate swell failures developed was also noted on the Wadesmill bypass (Hawkins, 2013).

1.6.3.2 Compaction

Where sulphate swell failures have developed, there have been suggestions the treated material may have been too dry (i.e. dry of the OWC for compaction). ISC suggest that such material would provide an enhanced drainage pathway for water to infiltrate the material and cause ongoing swell. This drainage pathway would worsen any general site drainage issues (as noted in Section 1.6.3.1). Hawkins (2013) evidences that both the M40 and Wadesmill bypass failures were undertaken during periods of drought and higher than average temperature. Hawkins (2013) suggested that the dry conditions would have caused real difficulty in maintaining the fill material to the wet of OWC condition required by the specification.

1.6.3.3 Fill material mineralogy

Lime treated fill materials which do undergo a high degree of sulphate swell are considered likely to also be of high plasticity and the industry would typically associate a high risk of sulphate swell with certain geological formations, e.g. Lias Clay. Where >1% TPS fill materials are of low plasticity and / or significant granular content, the risk is perceived as much lower.

1.6.3.4 GGBS

ISC’s working experience was generally in support of reports that use of GGBS as a secondary binder reduces the risk of sulphate swell (Section 1.6.2). However, ISC were involved with a
sulphate swell failure, where a lime and GGBS treatment had been used to stabilise a high plasticity and high TPS clay fill. ISC had followed the Britpave (2005) recommended working method for the treatment of high TPS materials and laboratory soaked CBR’s had not indicated a high swell risk. However, the sulphate swell failure of the treated material developed over the winter period following the stabilisation works.

1.6.3.5 Research requirements

At the outset of this research project, the sponsors sought increased knowledge on how sulphate swell develops under site conditions and whether the swell risk of treating high TPS materials could be more clearly defined and managed. As there was concern over the validity of using the soaked CBR test to quantify sulphate swell risk, the study would ideally develop or adapt a test to determine the site risk of sulphate swell. With improved knowledge and means of assessing risk, a routine approach to the SLS of materials with a high TPS may become acceptable within specifications.

1.7 THESIS STRUCTURE

The thesis has seven main chapters and three published papers which detail the research work undertaken. The content of each chapter and suggested links to the publications (appendix A-C; Table 1.3) are summarised below.

Chapter 1 describes the process of lime stabilisation and influence the HA specification has upon lime stabilisation works in the UK. Sections 1 and 2 in Paper 1 (Appendix A) provide further context of lime stabilisation in the UK. Incidence of sulphate swell failures on high profile UK projects are used to introduce sulphate swell as an area where the sponsor companies seek further knowledge.
Chapter 2 sets out the study aim and objectives and describes work packages used to deliver these. It is noted here that a significant challenge for this study (and the industry) is interpreting whether the findings of existing laboratory studies will be significant at the site scale. Therefore, along with the objectives set, the work packages were defined around this challenge. Section 3 in Paper 1 provides further detail on the additional processes applicable at the site scale and may aid with understanding this challenge.

Chapter 3 reasons the methodology followed and the laboratory experimental considerations observed during execution of this work. The options for replicating the site process and environmental conditions within a laboratory study are compared as part of this. The laboratory preparation method used to replicate the site process of lime stabilisation and as followed throughout this study is detailed in section 2 of Paper 2.

Chapter 4 reviews the literature on lime stabilisation processes and sets out the need for experimental work to investigate the effectiveness of these processes at a scale relevant to site applications. The need for this study to undertake site relevant experimental work on clay soils with and without high values of TPS is explained. With respect to literature review, this chapter adds further detail to the literature summarised in Section 4 of Paper 1, therefore section 4 of Paper 1 is a useful introduction to this chapter.

Chapter 5 details and discusses the site relevant laboratory experimental work undertaken on a lime stabilised clay soil without high TPS. The study investigates how larger clay clods influence development of the lime stabilisation processes through a compacted specimen. Detailed consideration of these processes identify how engineering properties at the site level may differ from those identified in laboratory studies which do not use large clods. Paper 2 and Paper 3 report on two different aspects of this experimental work. The chapter text is used to explain the overall experiment, link and present further evidence of the key findings noted in the two papers and to contextualise findings with the study aim.
Chapter 6 details and discusses the site relevant laboratory experimental work undertaken on lime stabilised clay soils with a high TPS and how this was developed from the preceding test programme. The chapter includes a review of literature on the chemistry of sulphate swell and literature reports on how the degree of swell may be reduced by adapting the treatment (section 4.3 in Paper 1 extends the site related discussion of this). A new test method to assess sulphate swell under site relevant conditions is established and used as part of the experimental programme investigating methods to reduce sulphate swell. The experimental work in this chapter has not been published yet and the text fully explains the experimental procedure, before discussion and interpretation of key findings relevant to the project aim.

Chapter 7 summarises the contribution of this EngD to practice and academia, key implication for the sponsor’s and the wider industry and makes recommendations for future research.

Appendix A-C contain the full contents of the three papers referred to within the above thesis overview and which are cross reference at relevant points of the main text. These papers should be read in conjunction with the thesis, so that the value of each publication can be understood in context with the overall research programme. An outline of each paper is provided in Table 1.3.

<table>
<thead>
<tr>
<th>Reference</th>
<th>Title</th>
<th>Publication</th>
<th>Status</th>
<th>Synopsis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Paper 1 Appendix A</td>
<td>Lime stabilisation for earthworks: A UK perspective</td>
<td>Proceedings of the ICE: Ground Improvement</td>
<td>Published (peer reviewed)</td>
<td>Review paper summarising key literature relevant to site practice of SLS in the UK</td>
</tr>
<tr>
<td>Paper 2 Appendix B</td>
<td>Nucleation centres in lime stabilised soils</td>
<td>ISSMGE International Symposium on Ground Improvement (Brussels, Belgium)</td>
<td>Published (peer reviewed) conference proceedings</td>
<td>Laboratory study investigating the role of finely ground limestone in the nucleation of pozzolanic reaction products in specimens prepared from large clay clods</td>
</tr>
<tr>
<td>Paper 3 Appendix C</td>
<td>Lime diffusion and implications for lime stabilisation practice</td>
<td>94th Meeting of the Transportation Research Board</td>
<td>Published (peer reviewed) conference proceedings</td>
<td>Laboratory study investigating how processes at both microscopic and field scales influence the engineering properties of a lime stabilised material.</td>
</tr>
</tbody>
</table>

Table 1.3 Summary of EngD publications
2 AIM AND OBJECTIVES

2.1 AIM

Based on the background to this research the overall project aim was to determine the site relevant lime stabilisation processes which cause sulphate swelling and develop an approach to define the risk of this failure.

2.2 OBJECTIVES

To achieve the aim, the below objectives were formulated

- **Objective 1 (O1)**. Establish the state of lime stabilisation scientific knowledge in reference to UK practice;

- **Objective 2 (O2)**. Identify the processes which lead to either beneficial or deleterious outcomes from lime stabilisation applications;

- **Objective 3 (O3)**. Develop a site relevant test method to appraise the risk of sulphate swell;

- **Objective 4 (O4)**. Determine the effectiveness of adaptations to lime stabilisation working methods in limiting the degree of sulphate swelling.

2.3 RESEARCH PROGRAMME

A key aspect of the study was to create outputs of site relevance. From this fundamental start point, the objectives (O1-O4) were established to build up knowledge in a systematic manner that would then achieve that aim. This section describes how four work packages (WP1-WP4) led to outputs that address the objectives.
WP1 involved the close familiarisation with sponsor company ISC’s working practices, specifically with tendering/specification review, SLS execution and laboratory and field testing. This led to familiarisation with how SLS is specified, executed and validated. While much of this familiarisation was achieved early, it was maintained throughout the study.

With a working knowledge of SLS established, a literature review process (WP2) was undertaken with intent to compare / contrast literature suggestions with practice. This literature review process provided much of the basis for Paper 1. At this stage it was identified that much of the existing literature was difficult to directly apply to site applications. Specifically, it was noted that the vast majority of research had been undertaken on laboratory specimens prepared using methods which do not reflect site practice (Bozbey and Garaisayev, 2010). This divergence from site practice typically comprised the prior drying and pulverisation of clay soils into very fine crumbs, e.g. <2mm, before the addition of binder and mixing water. Site operations do not have a drying stage and instead the material is rotovated at its in-situ water content (see Section 1.3). The soil pulverisation on site is therefore much less, e.g. clay clods of 1-40mm and results in a much more heterogeneous distribution of lime through the compacted soil body. Thus, it was suspected that different soil structures and physico-chemical processes would result from lime treated soils prepared with these different methods.

As the field scale influences were not clear, it became the initial experimental focus of the EngD to investigate how field preparation influences the development of engineering properties in lime stabilised clay soils. As the inclusion of sulphate swell reactions would add an extra complication and potentially cause a key process to be missed, the first experimental phase focused on a clay soil of low TPS content. This was undertaken with extensive experimentation into how the use of clods influence the micro-chemical and macro-mechanical processes in a lime stabilised soil body (WP3) and lead to the publication of Papers 2 and 3.
This study enabled a better understanding of the site relevance of the literature reviewed in WP2 and was also included within Paper 1.

Paper 1 included a summary of several literature suggestions on how the risk of sulphate swell may be reduced. However, it was not clear how these suggestions could be adapted into a site working method that would be successful in mitigating sulphate swell under field conditions (see Section 1.6.3.1). Furthermore, Paper 1 summarises that the existing laboratory test methods (e.g. soaked CBR) do not reflect the conditions that lime stabilised soils are exposed to on site (Notman, 2011). Thereby, the suitability of the existing test methods in quantifying risk of sulphate swell were under question. This led to a further experimental program (WP4) investigating which of the literature suggestions on methods to reduce sulphate swell risk were relevant under site conditions. This laboratory study included development of a new swell test which included provision for assessing field conditions which the soaked CBR test lacked (O3). As the findings from WP4 have not been published yet, they are reported and discussed in Chapter 6 (O4).

With successful execution of the described research programme it has been possible to address the aim and objectives (Figure 2.1) and provide the sponsors with an enhanced understanding of the risks associated with the SLS of high TPS soil (as summarised in Chapter 7).
Figure 2.1 EngD Thesis research map
3 ADOPTED METHODOLOGY

3.1 METHODOLOGICAL CONSIDERATIONS

Research projects may follow methodologies intending to produce either qualitative or quantitative data to address the chosen aim. Quantitative data is achieved through direct measurement, usually during experimentation. The quality of quantitative data may vary substantially depending upon the type of experimentation (Table 3.1). Very high quality datasets may be achieved from a trial with few or precisely controlled and monitored variables, using high sensitivity and calibrated equipment and, as repeated on numerous sub-samples of the same specimen. Thus, where the opposite is true, the quality and reliability of the data will reduce. Sometimes technological or practical restrictions mean it is not possible to make precise numerical measurements of data. Instead, it may be possible to correlate changes in the measured values with proportional changes in the analysed variable, resulting in a semi-quantitative analysis. Notwithstanding, semi quantitative data should still be repeatable and the reliability of the data will increase when proportionate correlations are upheld with further repeat tests.

<table>
<thead>
<tr>
<th>Data quality range</th>
<th>Low</th>
<th>High</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of Experimental variables</td>
<td>Numerous</td>
<td>One</td>
</tr>
<tr>
<td>Control over / monitoring of experimental parameters</td>
<td>None/limited</td>
<td>Precise</td>
</tr>
<tr>
<td>Suitability of Measurement equipment</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Accuracy / calibration of measurement equipment</td>
<td>Limited</td>
<td>Precise</td>
</tr>
<tr>
<td>Repeatability (number of sub-samples)</td>
<td>Low</td>
<td>High</td>
</tr>
</tbody>
</table>

Table 3.1 Experimental influences upon quantitative data quality.
Qualitative data are often obtained through consideration of the experiences and / or opinions of people. The methodology may involve conducting questionnaires, interviews, or review of verbal/written accounts to identify common responses. Where substantial responses are obtained, these may be analysed to determine the statistical relevance of response patterns. Challenges with achieving high quality qualitative data relate to ensuring questionnaires or interviews remain objective by avoiding question styles that may influence the respondent / interviewee. Further challenges may occur when the respondent’s employment or background may prevent provision of a neutral answer, again potentially increasing the subjectivity of these data.

3.2 METHOD

During the early stages of this EngD research, it was noted that qualitative data may assist with addressing the study objectives. Discussions with the industry sponsor had provided anecdotal evidence of the potential site triggers of sulphate swell (Section 1.6.3) which were used to help define the study aim and objectives. It would have been possible to extend upon this by interviewing key figures involved in the execution of both unsuccessful (e.g. M40) and successful lime stabilisation projects. This would potentially lead to identification of further lessons learnt from these failures that are not apparent or clear from existing literature. It would also have been possible to gauge the opinion of researchers and practitioners on how different sized clods appear to influence engineering performance. However, in addition to the potential subjectivity of quantitative research, there were some further challenges with identifying appropriate individuals to interview. Firstly, lime stabilisation failures tend to result in litigation between the involved parties, which may cause an unbalanced viewpoint or defensive interviewee. Secondly, there were very few individuals that had both in-depth knowledge of
academic research and the working practices of SLS. With a very small selection of people to interview, it would be difficult to achieve a range and depth of high quality data.

Balanced against the potential benefit of pursuing qualitative data from interviews was the strong suitability of a quantitative testing programme in meeting the study sponsor’s needs. The desires of the study’s sponsors related to development of SLS treatment method to control the risk of sulphate swell (Section 1.6.3.5). For such a method to gain acceptance from the wider industry, the basis of the test method would need strong justification. Thus it was decided to use experimental programmes to provide quantitative data that would justify and support the proposed sulphate swell test and any adaptations to the SLS working method.

With a decision to undertake a quantitative test program to meet the project aim, there were then several key decisions relating to methodology. It was identified that most research studies into lime stabilised soil involved initial classification of the soil properties (e.g. water content, compaction and plasticity), followed by a means of assessing changes to engineering properties following curing and progression of the lime-clay processes. For the latter it was typical to see use of either unconfined compressive strength, triaxial tests or index tests, e.g. California Bearing Ratio (CBR). For studies considering volume stability (including sulphate swell), the curing period would often include a phase where the sample is immersed (either fully or partially) in water and volume change measured. With physical testing complete, tests on the destroyed stabilised soil specimens would typically be analysed for changes e.g., to mineralogy, water content or plasticity, with intent to linking any measured changes with the engineering behaviour of the test specimen. A similar approach for this EngD study was considered beneficial to the project aim and consistency with this usual approach would assist widespread acceptance.
With an overall laboratory methodology determined, it remained necessary to establish a reliable method to replicate the field preparation used in SLS. Thereby the key factors to consider for the preparation method were:

- Site relevance of prepared specimens;
- Control of variables.

For the site relevance aspect, a suitable method was primarily considered against how closely the approach would replicate on site mixing to produce a site realistic distribution of clay clods with a surface coating of binder (see section 1.3). However, use of large clods (e.g. 20mm diameter) of natural clay soil would provide a potential conflict with the quality of data attainable. As noted in Table 3.1, where experimental variables increase, the potential for an associated variation in the measured output may coincide. Even before the added complication of a chemical binder, the heterogeneity of a natural clay soil (e.g. variations in water content, mineralogy, particle size distribution and soil structure) may lead to substantial changes in the soils engineering properties, sometime over very small distances e.g. centimetres (Mitchell & Soga, 2005). Thereafter, further variables e.g. environmental conditions and compaction quality, may also influence experimental output. As both site relevance and control over variables were considered of equal importance, four possible preparation methods (as summarised below) were reviewed for the value contributed to each (Table 3.2):

1. **Full field preparation**: Specimens formed from field stabilised material and after full compaction, e.g. using cutter mould or trim excavated blocks. Samples returned to laboratory for controlled curing and later analysis:

2. **Partial field preparation**: Samples of lime treated clay clods recovered after the SLS mixing and mellowing phases, but before field compaction. Specimens
formed in the field by manual compaction into a mould. Samples returned to
laboratory for controlled curing and later analysis:

3. **Lab with site realistic preparation.** Clay clods are created by hand pulverising
a carefully selected ‘as dug’ clay soil. Binder and mixing water is mixed around
cloids using a method which replicates site mixing. Specimens created using
automatic compaction. Curing and later analysis are laboratory controlled.

4. **Lab with artificial preparation.** The clay soil is first oven dried and crushed
to <500µm. The lime binder is closely mixed into the dry soil before addition
of mixing water to produce a relatively homogenous distribution of lime – clay
soil to the desired water content. Specimens created using automatic
compaction. Curing and later analysis are laboratory controlled.

Thereby, it was concluded that lab preparation using a site realistic method was the best
compromise between site relevance and control of variables. As the greatest scope for
inaccuracies between specimens related to heterogeneity in the source clay and differences in
clay clod sizes, these were identified as areas requiring the closest attention when finalising the
detailed preparation (See Sections 5.5 and 6.2.4).
Enhancing the understanding of lime stabilisation processes

<table>
<thead>
<tr>
<th>Variable</th>
<th>Specimen Preparation (see section 3.2 for detail)</th>
<th>Full field</th>
<th>Partial field</th>
<th>Lab – site realistic</th>
<th>Lab - artificial</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Variable 1</strong></td>
<td>Clay soil heterogeneity</td>
<td>×</td>
<td>✓</td>
<td>✓</td>
<td>✓✓✓✓</td>
</tr>
<tr>
<td></td>
<td>Low control; limited observation</td>
<td>✓</td>
<td>Minor control; Spot samples</td>
<td>Careful selection of clay; Blending of batches</td>
<td>Well blended batches; Consistent water content</td>
</tr>
<tr>
<td><strong>Variable 2</strong></td>
<td>Clay clod size</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
<td>✓✓✓✓</td>
</tr>
<tr>
<td></td>
<td>Minor control, spot checks only.</td>
<td>✓</td>
<td>Minor control Measurable</td>
<td>Moderate control between batches; measurable</td>
<td>Close Control</td>
</tr>
<tr>
<td><strong>Variable 3</strong></td>
<td>Binder mixing</td>
<td>✓ ✓</td>
<td>✓ ✓</td>
<td>✓ ✓ ✓</td>
<td>✓ ✓ ✓</td>
</tr>
<tr>
<td></td>
<td>Site plant mixing around clods</td>
<td>✓ ✓</td>
<td>Site plant mixing around clods</td>
<td>Thorough hand mixing around clods</td>
<td>Machine mixing around fine particles</td>
</tr>
<tr>
<td><strong>Variable 4</strong></td>
<td>Compaction</td>
<td>✓ ✓</td>
<td>✓ ✓ ✓</td>
<td>✓ ✓ ✓</td>
<td>✓ ✓ ✓</td>
</tr>
<tr>
<td></td>
<td>Field compaction to consistent method</td>
<td>✓ ✓ ✓</td>
<td>Manual compaction into mould</td>
<td>Machine compaction into mould</td>
<td>Machine compaction into mould</td>
</tr>
<tr>
<td><strong>Variable 5</strong></td>
<td>Curing Temperature</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
</tr>
<tr>
<td></td>
<td>Full control (recovered specimen transported to lab for curing)</td>
<td>✓ ✓ ✓ ✓</td>
<td>Full Control (compacted specimen transported to lab for curing)</td>
<td>Full Control</td>
<td>Full Control</td>
</tr>
<tr>
<td><strong>Variable 6</strong></td>
<td>Post cure soaking</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓ ✓</td>
</tr>
<tr>
<td></td>
<td>Full Control (recovered specimen transported to lab for soaked curing)</td>
<td>✓ ✓ ✓ ✓</td>
<td>Full Control (specimen transported to lab for soaked curing)</td>
<td>Full Control</td>
<td>Full Control</td>
</tr>
<tr>
<td><strong>Variability total</strong></td>
<td></td>
<td>12/24</td>
<td>15/24</td>
<td>19/24</td>
<td>24/24</td>
</tr>
<tr>
<td><strong>Site relevance total</strong></td>
<td></td>
<td>✓ ✓ ✓ ✓</td>
<td>✓ ✓ ✓</td>
<td>✓ ✓</td>
<td>✓ ✓</td>
</tr>
<tr>
<td></td>
<td>20/24</td>
<td>17/24</td>
<td>15/24</td>
<td>4/24</td>
<td></td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td></td>
<td>32/50</td>
<td>32/50</td>
<td>34/50</td>
<td>28/50</td>
</tr>
</tbody>
</table>

Table 3.2 Evaluation of four preparation methods for EngD experimental work. Each option assessed for site relevance and potential for variables to influence the quality of quantitative data.
4 RESEARCH UNDERTAKEN: LITERATURE

This chapter sets out the key literature reviewed and explains how considerations sourced from literature and industry working were used to define the scope of the subsequent laboratory research work.

4.1 LITERATURE FINDINGS

The primary focus of the literature review was to understand how the lime-clay soil processes change the engineering properties of the treated and compacted fill under site conditions. For this it was important to first understand what was already known about how favourable and unfavourable engineering behaviour may result from these processes. The major findings of the literature review were developed into Paper 1, although to set out the approach to the laboratory program it is important to include and add to this key information in the main text. This chapter specifically adds further detail (to that in Paper 1) of how the pozollanic reaction products develop at the microscopic scale (section 4.1.1.3). This extra detail is important to aid understanding of how the outstanding questions to be addressed with the laboratory experiment were defined in Section 4.2).

4.1.1 THE LIME – CLAY SOIL REACTIONS

This initial discussion of the lime-clay reactions assumes the added lime is in close contact with the reacting clay component and ignores any requirement for the lime constituents to first migrate into position. The latter complication is added by field applications of lime stabilisation which feature large clay clods and was the key focus of the experimental work in this study.
Enhancing the understanding of lime stabilisation processes

4.1.1.1 Lime

Lime binder may refer to quicklime (CaO) or hydrated lime (Ca(OH)\(_2\)) and may be provided in various forms, i.e., pellets, flakes, powder or as particles suspended within a water slurry. Each variant of lime binder is produced to different physical/chemical criteria (e.g., as categorised in BSI, 2006) and may suit a specific soil stabilisation application better than another type/form (see section 4.1.1.2). The sequence of lime-clay reaction must first be discussed, before implications of lime binder type can be considered further.

4.1.1.2 Drying

Substantial drying is only applicable where quicklime is used as the binder, removing excess water from the soil system through the action of “slaking” the quicklime (CaO) to calcium hydroxide (portlandite; Ca(OH)\(_2\)) as per Equation 4.1.

\[
\text{CaO} + \text{H}_2\text{O} = \text{Ca(OH)}_2 + \text{heat} \tag{Equation 4.1}
\]

This combines 32% of the quicklime’s initial mass with water molecules to cause an increase in volume (potentially providing deleterious expansive stresses if in a confined environment). The heat generated by equation 4.1 is 17x10\(^9\) Joules per kg of CaO which causes evaporation to further reduce the water content (Greaves, 1996). Therefore, the influence is principally one of drying out the material. The slaking reaction is described as violent (Sherwood, 1993) and also imposes an associated immediate expansion. Accordingly, the reactivity of the quicklime should be suitable to the intended use. If a site is water logged, the use of very reactive quicklime powder (i.e., conforms with category 1 of Table 1 in BSI, 2006) can result in thermal plumes with potential to uplift dust particles and cause an environmental nuisance. Thus, for some scenarios it may be relevant to use quicklime of lesser reactivity (e.g., flakes/pellets which
conform with category 2 of Table 1 in BSI (2006). However, as it is important to ensure slaking is complete prior to final compaction and avoid uncontrolled expansion (Boardman and Glendinning et al., 2001) it is more usual for site processes to use category 1 quicklime. The heat from equation 4.1 also accelerates the subsequent cation exchange process (Holt and Freer 1998; section 4.1.1.2)

### 4.1.1.3 Cation exchange

The alteration of the clay-soil structure mainly involves the calcium ions and is regarded as a rapid cation exchange process occurring on the surface of clay particles. Clay particles typically exhibit surface charge imbalances and the negative charges are balanced by hydrated cations. Accordingly, individual clay particles are surrounded by adsorbed water in the diffuse double layer arrangement (Van Olphen 1977). The thickness of the diffuse double layer is controlled by several factors (Reeves et al., 2006) although the valence of the charge-balancing cations has primary influence. Incoming divalent cations exert a greater attractive force towards the clay particle surface than any monovalent cations (which are common to natural clay soils, e.g. K+ or Na+). This balances the clay surface charge with fewer hydrated cations and the thickness of the diffuse double layer shrinks in response (Bohn, 2002). As the diffuse double layers shrink, the electro-static charges on adjacent clay particles interact to a greater extent. Opposing negative charges of parallel aligned (face to face) clay particles are repelled and reconfigure to promote a flocculated, positive / negative charge (e.g. edge to face) arrangement (Figure 4.1). This causes silt sized aggregations of clay particles to group together (Bell, 1996) and two influences on the clay soil structure are suggested; an increase in micro-porosity, *intra*-aggregate to the flocculated particles Figure 4.1c); and a change to the meso-porosity, *inter*-aggregate to the flocculated particles (Figure 4.1d). This reduces the effective surface area of clay minerals in contact with the inter-aggregate pore water accounting for much of the
Enhancing the understanding of lime stabilisation processes

immediate change in physical properties of the clay soil associated with lime improvement (Bell, 1988):

- Reduced plasticity and shrink / swell;
- Promotion of brittle / friable behaviour
- Increased permeability.

As the described change in soil structure is dependent upon a suppressed diffuse double layer it might be expected that the effects could be reversed through suspension within water. However, this is not evident and the aggregations persist, even when reworked during classification tests, e.g. particle size distribution and Atterberg tests. This resilience is caused by the rapid growth of some pozzolanic reaction products (Diamond and Kinter, 1965). These early formed reaction products localise around points of contact (e.g. edge / face) between clay particles within the flocculated structure (Figure 4.1c). This discrete strengthening at point of contact explains how formation of a very small quantity of reaction product provides resilience against dispersion (Diamond and Kinter, 1965).

Figure 4.1 Changes to clay soil structure due to cation exchange (from Paper 1).
4.1.1.4 Pozzolanic reaction

The stabilisation of a soil occurs through growth of a cementitious product and is achieved by harnessing the pozollanic potential of clay minerals. A pozollan is a material possessing the property to react and harden upon mixing with water and Calcium Hydroxide (Ca(OH)$_2$) (Hewlett 1998). Therefore, the reaction primarily occurs between calcium, silica, alumina and water and the new minerals are similar in composition to those formed through hydration of Portland cement e.g. Calcium Silicate Hydrates (C-S-H) and Calcium Aluminate Silicate Hydrates (C-A-S-H). Some reaction products have been described as crystalline (Diamond and Kinter, 1965), although without use of special preparation techniques (e.g. high temperatures and / or additional chemical treatments) a poorly crystalline or amorphous gel product is considered more likely (Arabi and Wild, 1986).

The composition, growth rate and meta-stability of the reaction products formed by the lime-clay soil reaction is necessarily driven by the below key factors (Little et al., 2010)

- Competing minerals with preferred energetic;
- Reaction kinetics;
- Interference of other ions within the soil system;
- Amount of available reactants.

Therefore, the environmental conditions (e.g. pH and temperature), clay mineralogy (see section 4.1.1.3.2), presence of deleterious compounds (e.g. sulphate and certain organics), quantity of added lime are some of the key factors to consider.

Before deleterious reaction products resultant from sulphate activity are considered in section 4.1.2, it is first important to consider what is known about how the favourable pozzolanic reactions develop. This will help understand what changes may be associated with the change from an ameliorative to a deleterious process.
4.1.1.4.1  **Pozzolanic reaction mechanism**

The following summarises some key literature on the pozzolanic reactions, reviewed in approach to the initial experimental works. The intent of this summary is to identify the types of processes which may occur and to aid considerations of how these beneficial reactions may be encouraged.

Sloan (1964) used electron microscopy to examine the influence of calcium hydroxide and sodium hydroxide upon flakes of kaolinite within an aqueous suspension. Micrographs illustrated that both hydroxides instigate dissolution of the edges of kaolinite flakes. This attack was noticed after 24 hours and continued throughout the 15 day experiment. The presence of new phases were identified after 4 to 7 days of curing in samples treated with both hydroxides, and were described as “layer lattice silicate” removed from the dissolved kaolinite. For the calcium hydroxide only, growth of another new phase was noted to nucleate aligned with the edges of kaolinite after 48 hours. By 72 hours, this product was then observed detached from the kaolinite, having formed clustered aggregations measuring up to 1µm. This crystalline phase, was identified from a diffraction pattern and tentatively identified as Prehnite (a C-A-S-H). A final point of interest from this study was the described alteration of crystalline kaolinite to “silicate relicts” of no/very low structural order. This was interpreted as the influence of the high pH breaking covalent bonds within the kaolinite.

Bell (1996) reported a study on pure clays of kaolinite and montmorillonite. (assumed to be a Ca–montmorillonite based on a reported W_L of 115%). Photomicrographs illustrated dissolution of kaolinite edges and a gel-like material coating the floccs, which was interpreted as a reaction product bonding adjacent clay particles together. Comparative montmorillonite samples were described as containing hazy patches of reaction product at the edges of clay particles.
Boardman et al., (2001) similarly studied pure kaolinite and Na-Bentonite. Findings concurred with Bell’s (1996) description of kaolinite dissolution, adding that the location was at the side of the pH dependent charge site. The authors noted that dissolution of bentonite resulted in a reaction product predominantly consisting of Ca and Si with only minor Al. This was attributed to the 2:1 mineral structure of bentonite and relative high supply of Si over Al. Comparatively, the kaolinite derived product consisted mainly of Al, with little Si, again reflecting the greater availability of Al from the edge sites. The authors considered that mineral dissolution was precursory to the precipitation of reaction product. They identified that no reaction product had developed after 7 days with only minor amounts noted after 175 days. This suggests very slow dissolution rates can apply to pure clays.

Conversely, Stocker (1975) reported that the dissolution of a predominantly Na Montmorillonite, natural clay did not have a limiting effect on the precipitation rate of the pozzolanic product. The new product, reported as a C-A-S-H, was considered to form a ‘reaction rim’ effectively blinding over the clay aggregations and restricting lime from diffusing deeper into the clay aggregations. This reaction rim was considered to support Diamond and Kinter’s (1965) interpretation of a rapid formed product (see section 4.1.1.3).

In a study on a lime treated illite rich red marl Arabi and Wild (1986) used high temperatures (75°C) and high (10-14%) hydrated lime addition to accelerate pozzolanic reactions. The reaction products were visible using Scanning Electron Microscopy (SEM) and noted as very fine (e.g. 1µm) lath like fibres coating the surface and edges of illite platelets. The fibre coating increased in size and coverage with prolonged curing, corresponding with sample strength increase. Transmission Electron Microscopy (TEM) was used for elemental analysis identifying the reaction product as an amorphous C-S-H gel of unusually low Ca/Si ratio; typically 0.5. It was proposed that the low Ca/Si ratio helped evidence the mechanism of lime clay reaction, as had the silicate layers (dissolved from the illite) been finely divided, the
C-S-H would polymerise with a higher Ca/Si ratio (≈0.8). Instead it was suggested the added lime caused only partial break down of the silicate layers and that these silicates were then modified by the lime to produce the C-S-H gel.

In an investigation using slurry’s of several natural clay soils treated with Ca(OH)₂, Wilkinson et al., (2010) report SEM images revealing the location of the reaction products. In a similar manner to that described by Arabi and Wild (1986) the growth of ≈1µm lath like C-S-H extended from the surface of clay particles and into void space or to an adjacent interface. Wilkinson et al. (2010) linked the growth of C-S-H across congruent interfaces with strength increases of the sample and other improvements to engineering properties. Wild et al. (1987) made similar observations, suggesting that the C-S-H gel filled in the inter-aggregate pore space, reducing permeability and increasing strength through ‘pore blocking’.

While the literature may not have been unanimous on the detail of the reaction process, it did provide two general mechanisms for consideration during subsequent investigations. The mechanisms may generally be grouped into those involving the topo-chemical alteration of an alumino-silicate host to form the new phase in situ (e.g. Stocker, 1975); or via a through solution processes, i.e. the dissolution of reactants from an alumino-silicate host, followed by transportation and then precipitation of the new phase at an alternative location (e.g Wild et al., 1987).

4.1.1.4.2 Clay mineralogy influence

The clay mineralogy is considered of particular importance to the rate at which the pozzolanic reactions result in strength increase (Bell, 1988). This is linked to a hierarchy of clay minerals where expansive clay minerals, such as montmorillonite, are recognised as providing the greatest rate of reactivity thus enabling maximum efficiency of pozzolanic reactions. The high reactivity of these expansive clay minerals is determined, among others, by their very high
surface area and the extra silicate layer within 2:1 minerals (Table 4.1; Elert et al., 2008; Ingles and Metcalf, 1972). These cannot be the sole factors, because Ingles and Metcalf (1972) consider the reactivity of illite to be less than that of kaolinite. In this regard, Bell (1988) suggested that not all the silicate in 2:1 clay minerals would be freely available, with illite and chlorite as examples where ionic bonding might restrict silica availability. Furthermore, Al-O covalent bonds are weaker than Si-O bonds and therefore aluminate supply will initially exceed silicate supply. However, as clay soils have a greater overall silica content, this supply rate will gradually reverse with on-going dissolution (Cristelo et al., 2012). The degree of crystallinity within the alumino-silicate source also has a high influence on bond strength and compared to well-ordered clay minerals, amorphous alumino-silicates rapidly dissolve (Duxson et al., 2007). This also explains why some industrial processed alumino-silicates, which have been subject to prior significant heat (e.g. GGBS), have little crystalline order and are rapidly activated by calcium hydroxides to produce substantial quantities of reaction product (Wilkinson et al. 2010).

<table>
<thead>
<tr>
<th>Clay Mineral</th>
<th>Layer structure</th>
<th>Surface area [m²/g]</th>
<th>Swelling properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolinite</td>
<td>1:1</td>
<td>10</td>
<td>None</td>
</tr>
<tr>
<td>Allophane</td>
<td>1:1</td>
<td>700-900</td>
<td>None</td>
</tr>
<tr>
<td>Smectite</td>
<td>2:1</td>
<td>800</td>
<td>Extensive</td>
</tr>
<tr>
<td>Illite</td>
<td>2:1</td>
<td>20</td>
<td>None / very little</td>
</tr>
<tr>
<td>Vermiculite</td>
<td>2:1</td>
<td>400</td>
<td>Limited</td>
</tr>
<tr>
<td>Chlorite</td>
<td>2:2</td>
<td>10</td>
<td>None</td>
</tr>
</tbody>
</table>

Table 4.1 Basic properties of common clay minerals (Rowell 1994))

### 4.1.1.5 Deleterious Mineralogy

As noted above the clay mineralogy, and the rate that reactants are brought into contact with the added lime, are linked to variations in the chemical composition of the reaction product. Furthermore, the abundant early supply of an alternative chemistry, e.g. SO₄²⁻ ions, may dramatically change the reaction product from an ameliorative into a potentially deleterious
mineralogy e.g. ettringite or thaumasite. An introduction to ettringite and thaumasite in lime-
is provided here before more detailed literature consideration relating to the development of
swelling in a lime clay soil system are included as part of the sulphate swell trial hypothesis
(Section 6.1).

4.1.2 ETTRINGITE AND THAUMASITE

Ettringite is a hydrous calcium alumina-sulphate mineral which precipitates under high pH and
sulphate conditions (Little et al., 2005). The basic formula for Ettringite is shown in equation
4.2. The trivalent metal Al$^{3+}$ may substitute with Fe$^{3+}$ or Cr$^{3+}$, whereas the oxyanion SO$_4^{2-}$
may substitute with CO$_3^{2-}$, SeO$_4^{2-}$ or CrO$_4^{2-}$. Other ionic substitutions are possible but unlikely at
the near surface. This is with the exception of silicate bearing Thaumasite (equation 4.3), which
may form a solid solution series with ettringite (Little et al. 2005).

\[
\text{Ca}_6[\text{Al(OH)}_6](\text{SO}_4)_3 \cdot 26\text{H}_2\text{O} \quad \text{Equation 4.2 Ettringite (Little et al., 2005)}
\]

\[
\text{Ca}_3[\text{Si(OH)}_6](\text{CO}_3)(\text{SO}_4) \cdot 12\text{H}_2\text{O} \quad \text{Equation 4.3 Thaumasite (Little et al., 2005)}
\]

The formation of either of these minerals requires supply of the reactants in the required molar
concentrations and conditions favourable to mineral growth. Ettringite formation is associated
with a high pH (>10) environment and while the mineral is reportedly stable over a wide
temperature range, e.g. 5-75°C, the solubility is temperature dependent (Perkins and Palmer
1999). In this regard Perkins and Palmer (1999) suggests that as temperature lowers, the
likelihood that ettringite will break down (as per equation 4.4) will decrease.

\[
\text{Ca}_6[\text{Al(OH)}_6](\text{SO}_4)_3 \cdot 26\text{H}_2\text{O} \rightleftharpoons 6\text{Ca}^{2+} + 2\text{AlO}_4^{-} + 3\text{SO}_4^{2-} + 4\text{OH}^{-} + 26\text{H}_2\text{O}
\]

\[\text{Equation 4.4 (Dissolution reaction of ettringite; Perkins and Palmer, 1999)}\]
The temperature dependence of ettringite solubility may also be influenced when specific reactants are in limited supply, e.g. where ettringite growth is controlled by the concentration and activity of \( \text{SO}_4^{2-} \). A chemical model proposed by (Hoglund, 1992) suggests that at 5°C, ettringite becomes stable at concentrations of \( \text{SO}_4^{2-} \) which are an order of magnitude lower than the concentrations required at a temperature of 20°C. Therefore, when the sulphate concentration is low, ettringite growth may increase as the temperature reduces. Further factors may be influential in limiting ettringite crystallisation e.g. the presence of chloride or carbonate (Hoglund, 1992). This suggests a relatively complex situation at temperatures relevant to the near surface environment in the UK and that ettringite solubility cannot be considered in terms of temperature alone.

### 4.2 OUTSTANDING QUESTIONS

The literature was useful in identifying possible mechanisms for the beneficial lime-clay reaction processes and noting that reaction products may be sub microscopic or up to of several microns in size. However, when considering the larger clods relevant to the field scenario, it was not clear how pervasive the development of these reaction products would be. Where studies showed micrographs (e.g. Arabi and Wild, 1986; Wilkinson et al., 2010) the reaction products could be seen on a broken surface, or periphery of a lime treated fragment of clay soil. Although, by the nature of the techniques used there could be no visual information on whether reaction products were also present deeper (e.g. 10µm, 100µm or 1000µm) behind this field of view. This causes specific difficulty with understanding how large clods would influence the development of pozzolanic reactions (whether beneficial or deleterious).
4.2.1 **Reactant Heterogeneity**

The fundamental issue for understanding pozzolanic reactions in the field scenario is how the initial heterogeneous distribution of lime over large clod surfaces, influences the chemical composition of the reaction products on or within the clods, and the timing of their growth. While the pozzolanic reaction may instigate rapidly when lime and the clay minerals are in intimate contact (e.g. Sloan, 1964), if lime transport through the clay clod were first required, presumably a delay, or induction period would precede? From the perspective of understanding sulphate swell this was especially important. Little et al. (2005) suggest that if all reactants (Equation 4.2) are immediately available, ettringite will form and reach a steady state within 150 hours. Furthermore, ettringite is only expansive if the water is sourced from outside the immediate system i.e. post compaction (Little et al., 2010). So if ettringite can be formed upfront in a homogenous system, the risk of long term volume change may be limited. Conversely, with the low solubility of sulphate, a heterogeneous distribution of reactants and a low permeability system, the risk of long term volume change from delayed ettringite growth may increase substantially (Little et al., 2010).

4.2.2 **Lime Migration / Degree of Pulverisation**

Much of the literature references in this section were included in the introduction and some of the discussion to Paper 3. The purpose of this text in Paper 3 was to demonstrate how the nucleation trial had increased knowledge of how lime stabilisation processes develop through large clay clods. The literature as repeated here has a different structure and the purpose is to demonstrate how at the start of this study, the small number of studies using larger clods limited understanding of how relevant the wealth of knowledge on pozzolanic reactions (Section 4.1.1.3) is to the site scale. Thus, this section uses the referenced literature to demonstrate the
need for the subsequent experimental work on lime migration in large clods, rather than for the discussion of it.

A key question with understanding reactant heterogeneity is: to what extent is lime able to migrate from point of application (i.e. the surface of a large clay clod) to areas deeper within the clay clod (e.g. to the centre of a large clod)? There are very few studies involving large clay clods, and even fewer that also considered whether it would then instigate pozzolanic reactions at the new location. The few publications that have investigated the influence of different clod sizes (such as Petry and Wohlgemuth, 1988; Bozbey and Garaisayev, 2010) report that the finest clod sizes (<5mm) are preferable for maximum strength and durability. Petry and Wohlgemuth (1988) suggested that the centres of larger clods might not react at all and that pozzolanic reaction was restricted to locations close to the clod periphery, although there was no micro-chemical investigation to substantiate this. The conclusion of Bozbey and Garaisayev (2010) was that field applications should seek to minimise clod size and avoid the inclusion of large clods. ISC’s experience suggests it is very difficult to achieve the very fine degree of pulverisation recommended, especially when treating clays of medium to high plasticity; as is the situation with the high TPS clay soils considered to be at high risk of sulphate swelling (see section 1.6.3.3).

In apparent contradiction to Petry and Wohlgemuth (1988) and Bozbey and Garaisayev (2010), Stocker (1975) reported that lime diffusion is capable of driving pozzolanic reactions into the core region of clods up to 40mm in size. Stocker (1975) used wet chemical analyses on inner and outer locations from 20mm and 40mm clay clods and concluded that lime was able to rapidly (within 3 days) migrate deep into the clod centres. Stocker (1975) also demonstrated that the lime present in both inner and outer clod locations caused the pervasive formation of C-A-S-H, which was termed ‘diffuse cementation’. Stocker (1975) was able to
correlate the progression of diffuse cementation with strength increases and durability to soaking water (after 10 days) which continued to increase for over 6 months.

With a lack of clarity from the limited information in the literature it was determined that the study experimental phase should directly consider how clay clod size influences the pozzolanic reactions.

4.2.3 DEVELOPMENT OF EXPERIMENTAL NEED

With the intent of the experimental work to investigate how pozzolanic reactions would develop in systems using larger clods, it was then decided to separate this into two test programmes. As there was a general lack of literature relevant to large clay clods, it was considered sensible for an initial programme to establish how beneficial pozzolanic reactions influence the engineering properties of a low TPS soil (Nucleation trial; Chapter 5). The second programme could then explore how the availability of sulphate may change the pozzolanic reaction products to form (presumably) expansive minerals (Sulphate swell trial; Chapter 6. It was ultimately intended to better understand how sulphate swelling develops under field conditions and to determine practical measures to reduce the risk of damaging heave (As per O2, O3 & O4; Chapter 2).
5  RESEARCH UNDERTAKEN: NUCLEATION TRIAL

This chapter is structured into subsections.

Sub sections 5.1 and 5.2 explain the intent of the trial and set out the literature basis for the hypotheses tested.

Sub section 5.3 provides an overview of the tests performed, including explanation of how this aligned with the industry approach to pre-contract laboratory testing (see section 1.3.1.4). Sub section 5.3 also summarises the content of the published papers developed from this phase of laboratory work, setting out how the remainder of the chapter cross-references these papers.

Sub sections 5.4 and 5.5 provide the detail of how the trial was executed and sub section 5.6 reports the results from the geotechnical tests.

Sub section 5.7 sets out the major findings of the work, using the key points established in the published paper to cross reference further evidence to strengthen the overall study findings.

Sub section 5.8 includes further evidence which was either not included within the publications, or has been set out in a manner which more clearly justifies the major findings of the work (sub section 5.7).

5.1  INTENT OF TRIAL

Initially, this trial intended to explore whether a high strength (as defined by BSI, 2010) profile of increased homogeneity may be produced from the heterogeneous distribution of lime over the surface of large clay clods.
Enhancing the understanding of lime stabilisation processes

The trial approach was formulated from a concept that the ideal scenario to achieve a homogenous distribution of reactants would be a clay / lime slurry intimately mixed together (as per Wilkinson et al., 2010). It was considered that, at the macro-scale, the cured, strengthened mass would act as a continuum of near uniform engineering properties and that this would provide durability against the softening effects of water ingress. It was considered that if this proved effective for a clay soil of low TPS, then it may also provide resistance to sulphate swelling in high TPS soils.

While the slurry ‘ideal’ may have homogeneity benefits, there would be a substantial practical issue with the very low shear strength of a slurry and the slow progression of lime-clay reactions (Wilkinson et al., 2010; Section 4.1.2 in Paper 3 discusses). Operation of earthworks plant typically requires the subgrade to achieve a minimum undrained shear strength of 40kN/m² or 2% CBR (Barnes, 2000), so this must be viewed as a practical minimum. Thereby, the trial intended to produce compacted samples with sufficient immediate strength to satisfy this minimum. This necessitated use of clay clods with the surface distribution of lime and the trial would explore:

1. Whether a wet of OWC condition would enable efficient lime diffusion deep into large clay clods (see reasoning in Section 5.2.1);

2. Whether finely ground limestone could act as nucleation centres to accelerate the growth of pozzollanic reaction products at the clod periphery and between adjacent clay clods (see reasoning in Section 5.2.2).

It was hypothesised that:

A. The deep and efficient diffusion of lime into the clods would cause the strength of the clods to increase in a relatively uniform manner (Barker et al., 2007; Stocker, 1975; section 5.2.1);
B. The addition of a finely ground limestone powder at the surface of the clay clods would provide a heterogeneous nucleation surface for the pozzolanic reaction products to grow from. This would accelerate the overall rate of reaction pozzolanic reaction and promote reactions at the surface of the clod, bonding adjacent clods together (see Section 5.2.2).

If these hypotheses proved valid, cured samples with a more heterogeneous suite of engineering properties may result as the clay clods themselves would be of relatively consistent strength and adjacent clay clods would be connected through cementitious bonding.

5.2 UNDERLYING THEORY FOR NUCLEATION TRIAL

5.2.1 LIME DIFFUSION / WATER CONTENT

Ion transport in clay soils is primarily driven by diffusion along electro-chemical gradients where ions redistribute to regions of low concentration Jungnickel et al., (2004). Although the diffusion process dominates in soils of high plasticity, advection becomes more influential with increased permeability, i.e. with permeability greater than $1 \times 10^{-9}$ m/s (Mitchell and Soga, 2005). Therefore, where soil plasticity is low or there is a significant granular component, advection may become the key ion transport mechanism. While ion diffusion predominantly involves the migration of charge balanced anion/cation pairs (Jungnickel et al., 2004), in a lime treated clay soil, the distribution of $\text{Ca}^{2+}$ occurs more rapidly and with wider coverage than the hydroxyl groups (Davidson et al., 1965). This suggests that lime does not diffuse as paired $\text{Ca[OH]}_2$ as might be anticipated in an advective system. Rogers and Glendinning (1996) agreed with Davidson et al (1965), reporting that the migration of hydroxyl groups from a lime source was limited to 20mm-30mm after 6 months, whereas $\text{Ca}^{2+}$ migration was greater and considered likely to continue with further curing. Rogers and Glendinning (1996) attributed this to the high reactivity of $\text{OH}^-$ with the surface of alumino-silicates causing the hydroxyl
diffusion to be self-limiting. Temperature is fundamental to the rate of diffusion, as increasing temperature is proportional to molecular excitement and kinetic energy for the ions to diffuse. Barker et al., (2007) highlight the importance of water in enhancing diffusion rates due to the thermal conductivity of water which transfers heat, increases molecular excitement and accelerates ion migration. Of note in this regard is Holt et al. (2000) who demonstrated that initially wet clods of reconstituted clay (lime added to 25-40mm clods at 1.2 x Wp) experience lime modification effects at a greater rate than dry mixed 2mm clods. This concurs with Barker et al. (2007) that increasing the degree of saturation in a clay soil enhances thermal flow and diffusion processes. Holt et al. (2000) utilised quicklime in their studies, the reaction heat from which, and especially in conjunction with the wet mix samples, may have further optimised lime modification rates. Bell (1988) agrees that a high water content is of paramount importance to an efficient lime stabilisation system, noting that if the system dries out while pH is still high, then the reactions will cease due to the curtailment of the transportation process.

5.2.2 **Nucleation Centres**

For a new phase to form from a solution, the concentration of reactants must exceed their solubility within the solution. Once saturated with reactants, the new phase may precipitate from the solution where this provides the solution with a lower energy state (assuming an exothermic reaction), although in order for this phase change to occur the activation energy of the reaction must be met. The activation energy may be considered as a threshold of potential energy that is higher than the energy state of the solution. Therefore, until the activation energy is met, a phase change cannot occur. Precipitation and growth of a new phase can only proceed from a surface that provides the lower energy required and this may associate with a homogenous or heterogeneous nucleation process. With homogenous nucleation, the lower energy surface is provided when sufficient molecules of the precipitant bond to form a nucleus...
meeting the critical radius. The critical radius associates with the point where the cluster has sufficient size to be stable and requires a sufficient number of molecular collisions to occur before they dissociate. Increasing temperatures will achieve the activation energy by providing kinetic energy and frequency to the collisions. Heterogeneous nucleation is where the critical radius is provided by particles of a solid phase of a different composition to the precipitant. As this negates the need for spontaneous collisions to provide the nucleus, a lower activation energy results and the inclusion of sufficient heterogeneous nucleation ‘seeds’ within a solution may cause a substantial increase to the rate of reaction i.e., a catalyst.

The potential value of nucleation centres within lime stabilised soils is set out in Section 1 of Paper 2 which explains how finely ground (0.5 μm – 1 μm) limestone is used for this purpose within Portland cement. More detail is provided in Paper 2 and a summary of the principle investigated is given here.

With the hydration of Portland cement, the mixing water has relatively low solubility and capacity to dissolve solutes from cement particles before super-saturation occurs. This prevents any further dissolution of the cement powder, until cementitious phases nucleate and precipitate from solution. The intermixing of a small percentage (5%) of finely ground limestone into the cement powder is effective in providing surfaces which encourage heterogeneous nucleation of the cementitious phases (Hawkins et al., 2003). The associated reduction in activation energy allows the new phases to grow at an increased rate, and the pore water is able to dissolve further reactants from the cement powder. Thus, the use of nucleation centres in Portland cement enables an increased rate of early strength gain (Winter, 2009).

The basis for this study was to investigate whether finely ground limestone could act as nucleation centres within a lime stabilised soil. With the addition of lime around a clay clod periphery, the alkaline hydrolysis of clay minerals (i.e. the breakdown of covalent bonds in water of a high pH to remove aluminate and silicate from the edge sites of clay minerals) may
be slow due to the pore water becoming rapidly saturated with ions of calcium, silicate and aluminate. Then, further dissolution of the clay minerals would be slow, until nucleation and growth of pozzolanic reaction products occurred. If finely ground limestone could act as nucleation centres, the precipitation of cementitious minerals and further dissolution of clay minerals may accelerate the rate of the pozzolanic reactions. This could potentially enhance the beneficial pozzolanic reactions at the field scale.

5.3 **NUCLEATION TRIAL METHODOLOGY**

5.3.1 **CORRELATION WITH INDUSTRY TESTING APPROACH**

A core aspect of the approach to laboratory testing was to use a method reflecting the approach ISC follow during pre-contract testing (see section 1.3.1.4). Of specific relevance is the use of:

- A preparation method using clay clods and a mixing process which simulates the site working method (see Section 5.5);

- The CBR index text as a primary assessment of improvements to engineering properties and durability to water ingress. While it is acknowledged that the CBR test is not a strength test in the strict sense, it does reflect the properties of stiffness, shear strength and deformation (Biczysko, 1996), hence an increase in CBR value is an indication in the beneficial promotion of one, or all of these attributes;

- The use of the Moisture Condition Value (MCV) test to align CBR results with the water conditioning of the material during compaction.

   To remain relevant to industry practitioners, the preparation and test regime used in the trial was similar. Furthermore, to better understand how values of failure stress/strain change with curing, the CBR tests were also complimented by 38mm quick undrained triaxial tests. To provide data of relevance to a pavement, or other near surface environment, use of low
confining stresses during triaxial testing was considered relevant. Throughout all tests 2.5kg ‘normal’ proctor compaction was used which is representative of the degree of compaction typically achievable on site.

5.3.2 EXPERIMENTAL OVERVIEW

The approach to laboratory testing followed the below general outline and further detail is given under relevant sub-headings:

- A test clay soil was classified and compaction / strength properties were determined over a range of water contents (section 5.4);

- Batches of the test clay soil were treated with lime only and lime + nucleation centres with compacted specimens created for CBR and triaxial testing following cure periods of 8, 32 and 194 days (section 5.5). These cure periods were selected as they are similar to the 7 and 31 days soaked CBR periods stated for the routine tests in HA74 (the exact number of days were not used due to these falling outside the university laboratory’s standard opening hours) and would reflect progression of the lime-clay reactions over the short and medium term. The longest cure period would provide opportunity for the pozzolanic reactions to substantially develop.

- Following curing, the treated specimens were tested to quantify the improvement in strength, stiffness or deformation properties (section 5.6). Samples were also obtained from the destroyed specimens for chemical / mineralogical testing (Sections 5.8.1-5.8.3).

Table 5.1 sets out the geotechnical tests (executed to the referenced British Standard) undertaken on the specimens produced. Some further tests using the techniques set out in Table 5.2 were undertaken on select samples (mostly from the 194 day cures).
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<table>
<thead>
<tr>
<th>Test name and standard reference</th>
<th>Natural Clay</th>
<th>After Mixing</th>
<th>Post cure*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture content (BS 1377:2)</td>
<td>✓ (7 No.)</td>
<td>✓ (5 No.)</td>
<td>✓ (3 No.)</td>
</tr>
<tr>
<td>Initial Consumption of Lime (BS 1924:2)</td>
<td>✓ (3 No.)</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td>Atterberg Limits (BS 1377:2)</td>
<td>✓ (3 No.)</td>
<td>✓ (2 No.)</td>
<td>✓ (2 No.)</td>
</tr>
<tr>
<td>pH (BS 1377:2)</td>
<td>✓</td>
<td>✓</td>
<td>✓</td>
</tr>
<tr>
<td>MCV (BS 1377-4)</td>
<td>✓ (1 No. test at 4 different water contents; see section 5.4.1)</td>
<td>✓ (3 No. per batch)</td>
<td>N/A</td>
</tr>
<tr>
<td>CBR (BS 1377-4 and BS EN 13286-47)</td>
<td>✓</td>
<td>✓ (3 No. Unsoaked 3 No. soaked)</td>
<td>✓ (3 No. Unsoaked 3 No. soaked)</td>
</tr>
<tr>
<td>38mm quick undrained Triaxial (BS 1377:7)</td>
<td>✓ (3 No. tests at 4 different water contents; see section 5.4.1)</td>
<td>✓ (3 No.**)</td>
<td>✓ (3 No.**)</td>
</tr>
</tbody>
</table>

* The same number of tests were undertaken on both nucleation and non-nucleation centre treated batches.

** Note further batches of the 38mm triaxial samples were produced after this initial phase of sample preparation (see section 5.5.2)

Table 5.1 Summary of standard geotechnical tests undertaken at the various stages of the nucleation trial.

Unless stated otherwise (e.g. triaxial tests) any reported results are average values from all tests.

5.3.3 CONTEXT OF THE PUBLICATIONS

Paper 2 and Paper 3 were developed to report results arising from the nucleation trial. It is recommended that each paper is read in full as the text in this chapter (particularly Section 5.7) is used to note how key points made in the papers link together. To aid clarity in understanding the key points from the papers, a brief summary of the content of Papers 2 and 3 is given.

Paper 2 details the site realistic preparation method used in the study and compares the results from test specimens with and without the nucleation centres after 8 and 32 days curing. Paper 2 noted some evidence that the specimens with nucleation centres had slightly superior strength, stiffness or deformation properties. Of potentially more significance, it was identified
Table 5.2 Summary of each type of further analyses undertaken on clod gradations recovered from the destroyed 194 day cured CBR specimens. Unless stated otherwise any reported results are average values from all tests.

that a sample structure, from the air voids between clay clods (inter-clod porosity), appeared to cause a significant reduction to these engineering properties. It was concluded that results from specimens cured for 194 days and some detailed analysis (i.e. the tests in Table 5.2) were required to investigate whether this specimen structure would remain a weakness as the pozzolanic reactions progressed. Furthermore, some extra batches for triaxial testing at the same cure periods were planned to further investigate how the sample structure influenced specimen failure. This additional data would be used to assess whether the nucleation centres were successful in increasing the pozzolanic reaction rate and bind adjacent clay clods together over a longer cure period (as hypothesised; section 5.2.2).
With the execution of the further testing, it became apparent that there was not a substantial difference in the engineering properties of specimens with and without nucleation centres (see Section 5.6). For this reason the focus of Paper 3 was placed on how use of large clay clods influences the engineering properties of lime stabilised clay soils. As the role of nucleation centres appeared insignificant, and to reduce potential for confusing readers of this paper, only the results from batches without nucleation centres were presented in Paper 3. A key finding of Paper 3 was that the first hypothesis of the experiment (see section 5.1) was valid and that conditioning the material to wet of optimum for compaction resulted in lime diffusion and pozzolanic reactions deep into the clods. Furthermore, the sample structure from air voids between the clay clods was confirmed as a weakness in lime stabilised specimens even after 6 months curing. It was also identified that the air voids between clods acted as a preferential pathway for soaking water, worsening this weakness.

To clarify where the main text of this chapter complements and expands upon the main points and data of Papers 2 & 3, the introduction to each sub-section will make these additions clear. Furthermore, Papers 2 & 3 include substantial discussion and interpretation of the mechanisms underlying the key points made and the chapter text will make cross references to the relevant sections of the papers where appropriate.

5.4 THE TEST CLAY

This section summarises efforts made to source and determine the properties of a relevant test clay for the nucleation trial. While some of the properties of the test clay were included in Papers 2 (Table 1 and Section 3.1 therein) and Paper 3 (section 2.1), this section specifically adds CBR and triaxial test data on the clay over a range of water contents. This is useful in indicating the range of strength, stiffness and deformation properties which associate with water conditioning of the soil alone. This is a useful baseline from which to compare and contrast the
improvements that the lime treated specimens provide with curing. The clay was also tested for total chemistry (XRF), mineralogy (XRD) and using thermal analysis, although these results are included in sections 5.8.1 to 5.8.3 (which investigate the chemical composition of the pozollanic reaction products).

### 5.4.1 The Ideal Test Clay

In consideration with the overall methodology to replicate site practicalities (Section 3.2), it was decided to utilise an ‘as dug’ material sourced from a suitable site.

From literature review, industry experience and data quality considerations, it was ascertained that an ideal clay soil for use in the nucleation trial would possess the below attributes:

- Medium to high plasticity;
- Free from organics detrimental to lime stabilisation;
- Water Content close to or ideally greater than the plastic limit (WP);
- Uniform properties (e.g. water content, plasticity and degree of weathering) throughout the bulk sample.

A source of clay which reasonably met the criteria was identified at a quarry site and the open access at this location permitted the clean, hand dug excavation of an approximately 1m x 1m block of the clay soil. This was called ‘Cauldon Clay’ after the location it was sourced and some mineralogical details on provided in Table 1 of Paper 1.

Prior to use of the Cauldon clay in any tests the material was hand pulverised and blended (as described in Paper 1; Section 2.2.1), so that every batch of the clay would contain an even distribution of 14-20mm and <14mm clay clods. This was an attempt to ensure that natural variations in properties, inherent with using a 1m x 1m block of clay soil (see Section 3.2) would be blended across the batches.
5.4.2  CLASSIFICATION OF CAULDON CLAY

The host clay soil was classified by undertaking the tests listed in Table 5.1. To assess the engineering behaviour of the natural material over a range of water contents, suites of CBR, 38mm undrained triaxial (3 x specimens extruded from a 1 litre proctor mould), MCV and water content tests were undertaken after conditioning the material to different water contents. To achieve the desired range of water contents, a blended batch of the pulverised Cauldon Clay was divided into sub samples with the water content in each sub-sample conditioned as below:

- Tested at natural water content;
- Air dried for 6 hours;
- Air dried for 22 hours;
- Addition of a measured quantity of distilled water and hand mixed.

It is noted that with favourable weather this approach to water conditioning a clay soil could be replicated on an earthworks site.

Following any air drying / water addition and to permit pore water pressures to reach a relatively steady state throughout the clay clods, the material was sealed in a sample bag for over 24 hours. Thereafter, the test specimens were created and tested and the results are presented in Table 5.3 to Table 5.5 ; Figure 5.1 to Figure 5.2. The primary relevance of these data are that it demonstrates the range of unsoaked CBR values (1.5-10.5%), undrained strength and stress/strain response achievable for this clay by water conditioning alone. Furthermore, with an unsoaked CBR of 3.8% and cohesion (by shear vane) of 47kN/m² the Cauldon clay is the type of material which in its natural condition would likely require lime treatment for inclusion in a pavement subgrade / foundation layer design (or similar). This confirms the relevance of using this material as a test clay.
Research Undertaken: Nucleation Trial

### Water Content (%)

<table>
<thead>
<tr>
<th>Sample Ref</th>
<th>W&lt;sub&gt;D&lt;/sub&gt;</th>
<th>W&lt;sub&gt;L&lt;/sub&gt;</th>
<th>I&lt;sub&gt;P&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cauldon 1</td>
<td>30</td>
<td>72</td>
<td>42</td>
</tr>
<tr>
<td>Cauldon 2</td>
<td>31</td>
<td>69</td>
<td>38</td>
</tr>
<tr>
<td>Cauldon 3</td>
<td>30</td>
<td>66</td>
<td>36</td>
</tr>
<tr>
<td><strong>Average</strong></td>
<td><strong>31</strong></td>
<td><strong>69</strong></td>
<td><strong>38</strong></td>
</tr>
</tbody>
</table>

Table 5.4 Atterberg tests on Cauldon Clay

### Initial Consumption of Lime values for Cauldon Clay

Table 5.5 Initial Consumption of Lime values for Cauldon Clay

![Figure 5.1 Plot of dry density vs water content for cauldon clay (data from CBR mould weights)](image-url)
Figure 5.2 Stress – strain plots from 38mm undrained triaxial tests on Cauldon clay. The legend indicates the specimen water conditioning and test cell pressures are shown within parenthesis.

5.5 LIME STABILISED BATCHES

The method used to replicate the site mixing process used in SLS is described fully in Paper 2; Section 2. As this experiment had two different powders to add, i.e., quicklime and then limestone, a minimum of two mixing stages were required and the method simulated a ‘two-stage’ mixing process in field applications (see Section 1.3.3).
5.5.1 **Lime Binders and Nucleation Centres**

Two different forms of lime binder were used in the trials. With the first mixing stage a high reactivity quicklime powder (Proviacal ST, supplied by Lhoist) was added at a quantity equal to the ICL value of 3.5% at the first mixing stage. Then a Lime slurry (Proviacal SLS45, supplied by Lhoist) was added at a quantity equivalent to adding a further 5% of the quicklime. Thus, the total amount of lime binder added was the equivalent of 8.5% of quicklime.

There were several reasons for using two different lime binder types and these reasons were based on literature and site experience. Quicklime was used in the first mix as it was considered the heat from slaking would assist with pulverisation (to assist with an improved grading to lessen air voids upon compaction) and potentially also weakening the covalent bonds in the clay minerals, thus accelerating subsequent pozollanic reaction (see Section 4.1.1.4.2). Lime slurry was used for the second mix to avoid excessive drying from limiting ion diffusion and diffuse cementation of the large clods (see 5.2.1).

For the nucleation centres, a finely ground (480m²/kg blaine) limestone powder (Betocarb 80-BT, supplied by Omya UK Ltd was used.

5.5.2 **Batches and Quality Control**

The samples created during the initial phase of the trial are summarised in Table 5.1 and the same approach to batch and sample reference introduced in Paper 1; Section 2.4 is followed in this main text:

Days cured_Test name (cell pressure)

e.g. 8_CBR_S_Nuc or 32_TRI(50)_L.

Where:

S = Soaked; L = lime only (without nucleation centres); Nuc = with nucleation centres
Comparison of results from MCV, shear vane (conducted on the MCV samples) and oven dry water content tests confirm that efforts to blend the host soil and follow a set method resulted in consistency during preparation of the initial batches (Table 5.6). The aim to achieve an immediate compacted shear strength >40kN/m² was also achieved (Table 5.6). These results also reveal that, compared with the lime only, batches with nucleation centres had a lower water content (average of 2.0% less). As discussed in section 4 of Paper 2, this may be attributed to the additional solid mass of limestone added to these specimens. This may have caused a slightly higher dry density at preparation, slightly increasing the immediate and early cure strengths of the nucleation samples.

<table>
<thead>
<tr>
<th>Batch</th>
<th>MCV</th>
<th>Water Content (%)</th>
<th>Shear strength by hand vane (kN/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8_L</td>
<td>8.9</td>
<td>38.8</td>
<td>N/T</td>
</tr>
<tr>
<td>32_L</td>
<td>8.8</td>
<td>38.8</td>
<td>58</td>
</tr>
<tr>
<td>194_L</td>
<td>9.1</td>
<td>38.1</td>
<td>58</td>
</tr>
<tr>
<td>8_Nuc</td>
<td>8.7</td>
<td>37.1</td>
<td>60</td>
</tr>
<tr>
<td>32_Nuc</td>
<td>9.3</td>
<td>36.4</td>
<td>65</td>
</tr>
<tr>
<td>194_Nuc</td>
<td>9.3</td>
<td>36.3</td>
<td>64</td>
</tr>
</tbody>
</table>

Table 5.6 MCV, water content and shear vane values of the initial batches.

Following testing of the 32 day cures, the influence of the sample macrostructure upon the triaxial test failure planes was identified for the first time (Section 3.3 in Paper 1 and discussed further in Section 5.7.4 of this chapter). At this stage it was decided to prepare further batches of samples for 38mm undrained triaxial tests to look at this influence in closer detail and a further 6 specimens (extruded from 2 No. 1 litre moulds) were created for each cure period. As CBRs were not required, it was possible to prepare all the material for the 8, 32 and 194 day cure periods using one batch only for lime and one batch only for nucleation centre specimens. For the further batch with nucleation centres, it was decided to add mixing water to compensate for the lower water content resultant from the added limestone powder. As a result the difference between the water content and MCV’s for the extra lime and nucleation
centre batches were marginal and closely comparable with the initial lime only batches (Table 5.6 and Table 5.7).

<table>
<thead>
<tr>
<th></th>
<th>MCV</th>
<th>Water Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8, 32, 194_L</td>
<td>9.2</td>
<td>38.6</td>
</tr>
<tr>
<td>8, 32, 194_Nuc</td>
<td>9.0</td>
<td>38.9</td>
</tr>
</tbody>
</table>

Table 5.7 MCV and water content values for the extra batches (all results are the average of at least 3 separate tests).

5.5.2.1 Curing Conditions

All curing was undertaken at 20°C, which is a temperature standard to many laboratory programmes of this nature. As lower average temperatures would associate with site conditions, it could be argued that it is more realistic to use lower temperatures e.g. 5-10°C for curing. As the test clay for this trial was of low sulphate content, the anticipated reaction products were C-S-H or C-A-S-H phases and it was not suspected that the composition would change substantially across the range of 5°-20°C. This is quite different from lime treatment of clays with high TPS, as the reduction of temperature below 15°C may be associated with the formation of the deleterious mineral thaumasite (see Section 4.1.2). Thus, for the low TPS trial it was considered the most likely effect of a temperature lower than 20°C would be a reduction to the rates of diffusion and reaction, rather than a change in chemical composition. So, laboratory curing at 20°C would still be relevant, however it would be expected that the rate of the reactions and improvements to engineering properties would take longer in the field.

For the soaked CBR’s the soaking arrangement was described in section 2.4 of Paper 2 and for ease of reference the soaking duration is repeated in Table 5.8.

<table>
<thead>
<tr>
<th>Total cure period (days)</th>
<th>8</th>
<th>32</th>
<th>194</th>
</tr>
</thead>
<tbody>
<tr>
<td>Days of total cure period soaked in water</td>
<td>4</td>
<td>28</td>
<td>28</td>
</tr>
</tbody>
</table>

Table 5.8 Soaking durations for the soaked CBR’s
5.6  **STRENGTH AND ATTERBERG TEST RESULTS**

The results for all the lime only specimens were presented and described in section 3 of Paper 3. However, other than the first batch of 8 and 32 day cures in Paper 2 (section 3.2) results from the nucleation specimen have not been discussed in the publications. The results from the specimens with nucleation centres are very similar to the lime only samples. Therefore, rather than repeat the description of the lime only results here, the results from the nucleation specimens are described and similarities to the lime only results noted therein. The CBR and triaxial test results for all specimens, including (for easy reference) the lime only specimens, are presented in Table 5.9 and Figure 5.3 to Figure 5.6.

5.6.1  **UNDRAINED TRIAXIAL TESTS**

As with the lime only values, the results from nucleation specimens identify that failure stresses increase substantially with ongoing curing, indicating an associated increase in the shear strength of the specimens. Other similarities with the lime only specimens are that for any given cure period, the failure stress of the nucleation specimens generally increases with confining stress. For the 194_Nuc specimens, tests at cell pressures of 100kN/m\(^2\) and 200kN/m\(^2\) were included to see if this association remained at higher confining stresses. The respective failure deviator stresses of 729kN/m\(^2\) and 747kN/m\(^2\) from these specimens provide a clear contrast between the tests from the 194 day cures undertaken at very low confining stress (i.e. cell pressures of 10kN/m\(^2\) and 20kN/m\(^2\) with respective failure deviator stresses of 544kN/m\(^2\) and 554kN/m\(^2\)) and demonstrate this trend. The increase in failure stress with confining pressure suggests a significant frictional component to the shear strength of the material and this may be from air voids within the specimen that are not saturated with water.

Notwithstanding, and as with the lime only specimens, there is significant inter-
specimen variability causing certain plots (e.g. 194_TRI(50)_Nuc; Figure 5.6) to deviate from this overall trend of increasing failure stress with confining pressure. Therefore, the influence of a sample macrostructure upon the failure planes of the nucleation samples is apparently similar to that described for the lime only specimens (this is discussed further in Section 5.7.4).

5.6.2 CBR

The CBR values also reflect a similar trend of improvement in strength, stiffness or deformation properties with curing (Table 5.9). While the CBR values for the Nuc specimens are consistently higher than the lime only specimens, the differences are slight (i.e CBR value approx. 1% greater at 194 days). When considering the dry density difference on preparation, this marginally higher value is not considered significant evidence of the nucleation centres providing strength benefit. For the soaked CBR’s, no sample swell was noted in any of the test specimens and soaking caused a reduction from the unsoaked CBR value. This reduction was greatest (approximately 20%) for the 32 and 194 day cures for which the soaking period were longest (28 days)

<table>
<thead>
<tr>
<th>Unsoaked CBR (%)</th>
<th>Soaked CBR (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Top</td>
<td>Bottom</td>
</tr>
<tr>
<td>8_CBR_L</td>
<td>9.7</td>
</tr>
<tr>
<td>32_CBR_L</td>
<td>14.2</td>
</tr>
<tr>
<td>194_CBR_L</td>
<td>21.1</td>
</tr>
<tr>
<td>8_CBR_Nuc</td>
<td>11.7</td>
</tr>
<tr>
<td>32_CBR_Nuc</td>
<td>15</td>
</tr>
<tr>
<td>194_CBR_Nuc</td>
<td>21.5</td>
</tr>
</tbody>
</table>

Table 5.9 CBR values for all cures of the lime only and nucleation samples.
Figure 5.3  Deviator stress vs strain plot for the initial batch of lime only triaxial tests (38mm unconsolidated undrained). Test cell pressures are shown within parentheses.

Figure 5.4  Deviator stress vs strain plot for the initial batch of Nuc triaxial tests (38mm unconsolidated undrained). Test cell pressures are shown within parentheses.
Figure 5.5 Deviator stress versus strain plot for extra batches of lime only triaxial tests (38mm unconsolidated undrained). Test cell pressures are shown within parentheses.
Figure 5.6 Deviator stress versus strain plot for extra batches of Nuc triaxial tests (38mm unconsolidated undrained). Test cell pressures are shown within parentheses.
5.6.3 **ATTERBERG TEST RESULTS**

Figure 5.7 presents the atterberg test results undertaken on bulk samples from the natural and cured specimens. Following 8 days cure values of $W_P$ and $W_L$ for lime only and nucleation samples increased significantly from natural and are considered to reflect the early lime clay reactions. Thereafter, there are only marginal changes in $W_P$ and $W_L$ by 194 days, which combine to result in an $I_P$ of 30%, reduced from the natural clay $I_P$ of 38%. The relevance of these results considered further in Section 5.7.2.

![Figure 5.7 Changes to Atterberg limits with curing](image)

5.7 **KEY FINDINGS**

This section sets out the key findings from the nucleation trial. Most of these have been discussed in Papers 2 and 3 and then applied to practice in Paper 1 and this section will initially summarise the findings from Papers 2 and 3. Where relevant, further evidence which may
include cross references to later sub-sections will be provided to strengthen or clarify the point made.

5.7.1 **Beneficial Pozzolanic Reactions**

Comparison of the tests undertaken on the natural clay and lime stabilised batches confirm that the lime treatment was successful in causing a substantial increase to strength. By 6 months cure the strengths were well in excess of values achievable from water conditioning of the clay alone and for all cures the peak stress was achieved at a lower strain (Figure 5.5 and Figure 5.6). This serves as a physical indication that the lime clay processes, including the pozzolanic reactions had developed with curing as expected from literature (see Section 4.1.1).

SEM-EDX (Section 4.3 of Paper 3) was used to identify that the chemical composition of the pozzolanic reaction product was C-S-H. The paper reports that micro-concretions of C-S-H up to 100\(\mu\)m across were evident at locations within 1 mm from the periphery of clods from 194 day cures. This is an important finding as literature suggested the maximum size of beneficial reaction products would be smaller e.g. \(\approx 1\mu\)m (see section 4.1.1.4.1). The preparation of specimens using cross sections through large clods (preparation detailed in Appendix D), an approach novel to this study, is the reason this discovery was possible. The SEM-EDX work was also used as a basis to suggest that the growth of sub-microscopic and gelatinous C-S-H around clay mineral aggregations was the main mechanism causing strength increase deep into the clay clods (see Section 5.4 in Paper 3). In support of the interpretation put forward, further detail on how the SEM-EDX analysis and interpretation was undertaken is presented in Section 5.8.1.

Section 4.3 of Paper 3 reports that XRD traces provide evidence the reaction products were not crystalline in nature and this was used as part of the evidence to suggest that the C-S-
H was a poorly crystalline gel. No XRD traces were presented in Paper 3 and these are included in Section 5.8.2 as further evidence of this interpretation.

Subsequent to the publication of Paper 3, thermal analysis was undertaken on samples from the 194 day cures (section 5.8.3). These results did not identify any clear difference between analyses on the untreated and cured specimens. It was suggested that this absence would still fit within the suggestion that C-S-H gel is finely disseminated in the pore space between clay mineral aggregations. This would be due to the mass percentage of C-S-H being very low compared with the host clay soil as the technique would not be able to detect C-S-H over background readings from the soil. Thereby the thermal analysis does not evidence for, or against the proposed pozzolanic reaction mechanism.

Section 5.4.4 in Paper 3 suggests limited changes to the Atterberg limits with long term curing (see Section 5.6.3 in this thesis) would support both the pozzolanic reaction mechanism proposed (see Section 5.7.2) and that the reaction products were gelatinous in nature. Paper 3 explains that the mechanism of the Atterberg test (i.e. using palette knives to mix the material with distilled water) would cause a C-S-H gel to solubilise into the constituent ions, exposing the underlying aggregation of clay minerals and allowing the usual plastic response. If the reaction product were of high crystallinity then a much greater reduction to the plasticity index would be expected. Therefore, where literature reports have associated very large reductions in plasticity with the successful progression of the pozzolanic reactions (e.g. Bozbey and Garaisayev, 2010) it may be that these reaction products were of higher crystallinity. Thus, changes to plasticity with curing was not a relevant indicator of the development of pozzolanic reactions evident in the 194 day cure specimens from this study.


5.7.2 **DIFFUSE CEMENTATION WITHIN CLODS**

The nucleation trial identified that diffuse cementation did occur deep into the clay clods up to 20mm in diameter and that this caused the measured improvements to specimen strength. To evidence that diffuse cementation did occur, it was first established in Section 4.3.1 of Paper 2 that by 194 days cure there had been substantial diffusion of ions of Ca$^{2+}$ and OH$^-$ into the centre of the 14-20mm clods. The hydroxyl groups caused the pH of the central region of the clods to increase to 11.8, a value well in excess of the pH 9-10 necessary to cause the alkaline hydrolysis of clay minerals. Sections 5.4.2 of Paper 2 explains how the hydrolysed silicate and aluminate would preferentially polymerise with calcium within the pore water to form C-S-H gel (as identified by the SEM analysis; Section 5.7.1).

Section 5.4.3 of Paper 2 states that the diffuse cementation process caused the strength of individual clods to increase at a relatively uniform rate. The basis of this was through observations of broken sections through individual clods, which permitted a visual and basic physical assessment of strength (i.e. using finger pressure / thumb nail indentation as relative indicators; as described in BSI, 2010). As part of this assessment it was apparent that the progression of the pozzolanic reactions between 32 and 194 days curing caused observable changes throughout the clay clods. Photographs of cross sections through representative clods reveal differences in surface texture between clods from 32 and 194 cures. The 194-day nodule had a solid appearance and was hard, evident from soil grater indentations, compared with the 32-day nodule (in this case broken by hand) which was stiff with a friable/brittle appearance (Figure 5.8). Through inspection of cross sections in this manner it was apparent that changes to clay clod strength were relatively consistent through the clods and the ‘soft centres’ suggested by (Petry and Wohlgemuth 1988) were not a feature of the larger clods in this study. Thus, it appears that compacting the specimens wet of the OWC (i.e. MCV of approximately 10)
Figure 5.8 Cross sections through 14-20mm clods from 32-day (left) and 194-day cures (right). Both clods were at ‘as tested’ water contents (not dried).

enabled the diffusion of $\text{Ca}^{2+}$ and $\text{OH}^-$ deep into the clay clods.

Section 5.4.3 in Paper 3 provides a closely referenced account of how the alterations to the clay minerals from the cation exchange process (as set out in Figure 4.1) would enhance the diffusion of $\text{Ca}^{2+}$ and $\text{OH}^-$ through the inter-aggregate porosity and this is summarised. It is explained how aggregation of the clay minerals would cause the majority of the clay mineral surface area to become internal to the aggregations. Thus, the surface area of clay minerals in contact with the inter-aggregate pore water (of high pH) would become much less and the resultant reduction in tortuosity through saturated pore channels would enhance ion migration. Evidence of the clay mineral aggregation was provided by comparing Environmental SEM (ESEM) images of the microstructure from the natural clay and lime cured specimens. The ESEM images (Figure 6 in Paper 3) show the natural clay with clay floccs separated by air voids, whereas the lime cured specimen shows 2-5$\mu$m aggregations of clay minerals interconnected by a pore water/gel like coating. Section 5.4.3 of Paper 3 concluded that while $\text{OH}^-$ migration can become self-limiting, the low surface area of clay mineral aggregations would limit this in the first instance, and wider migration of the hydroxyl groups through the saturated pore channels would result. With the migration of reactants throughout the clod, the
growth of C-S-H caused the diffuse cementation between clay mineral aggregations, resulting in relatively uniform strength increase within each clod.

It is worth noting that the mechanism proposed to explain diffuse cementation would also fit with some of the observations from the triaxial testing. The flocculation of clay minerals with cation exchange is associated with an increase to intra-aggregate porosity and with the reduction in thickness to the diffuse double layer, this pore space would remain air filled (see Figure 4.1). These intra-aggregate air voids may well explain the results from the triaxial tests, which demonstrate that the failure stress generally increases with confining stress (Figure 5.5 and Figure 5.6). However, as there was also air voids associated with the inter-clod porosity (Section 5.7.4) these may also be considered a potential explanation for this relationship.

5.7.3 **CLOD SURFACE BOUNDARY**

Section 5.1 of Paper 3 explains how during the mixing stage of lime stabilisation, the addition of lime to the surface of the clay clod creates a physico-chemical boundary and this is evidenced as an important concept for field applications of lime stabilisation. It is proposed that this clod surface boundary was caused by the rapid lime-clay reactions described in Section 4.1.1.3. The aggregation of clay minerals through cation exchange (Figure 4.1) is typically associated with an immediate and substantial increase in the plastic and liquid limits (Diamond and Kinter, 1965; Sherwood, 1993). Atterberg limits of $W_P = 47\%$ and $W_L = 85\%$ for the 8 day cures (Figure 5.7) provide an indication of how these values would increase at the clod surface during mixing. As these values are substantially higher than the 36.3-38.8\% (Table 5.6) water content of the samples during mixing, the absence of plastic behaviour between clods would not allow these to join together and an air gap would remain, even if compaction could force adjacent clods very close together. Therefore, unless the water content on mixing is very high (i.e. at least in excess of the lime modified $W_P$ and possibly close to the $W_L$) it seems unlikely that pore water
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in separate clay clods could become connected. The diffusion mechanism described in Section 5.7.2 relies upon a connected pore water system. Without connected pore water, it is not logical that the lime-clay pozzolanic reaction could infill or create a cementitious bond across this inter-clod porosity. An air gap between clay clods also explains why it was possible for clods to be recovered individually from each (post test) specimen extruded from the CBR 194 day cures (see Section 2.4 in Paper 3 and Figure 5.9 in this chapter). SEM analysis (Section 5.8.1.3) provides further evidence that whole clods were recovered individually from the CBR specimens. Figure 5.18 comprises an image from the SEM analysis showing that limestone particles (which were added to act as nucleation centres) are visible at the very edge of the clay clod recovered.

Figure 5.9 Representative clods from a 194_CBR_L retained on a 20mm sieve. The majority are 14-20mm in size, although several adjoined clod ‘blocks’ are also shown.

It is noted in Paper 3, Section 5.1 that the clod surface boundary appears to result in each clod having an internal system with no chemical transfer (e.g. Ca$^{2+}$ and OH$^-$ ions) between the clods. This boundary helps explain several key observations in this study i.e. inter-clod porosity and that lime diffusion caused pozzolanic reactions deep within (see Section 5.7.2), but not between clods. It was evidenced in section 4.2 of Paper 3 (and Section 5.7.4.2 of the thesis further discusses this) that the mixing process causes each clod to receive an amount of
calcium (from the lime) and water which is proportionate to the size of the clay clod. This supports the suggestion that calcium and water are not able to migrate between the clods and that the clods form a system of discontinuum. Section 5.7.4 explains in further detail how the heterogeneous distribution of lime and water, in conjunction with the inter-clod porosity resulted in a non-uniform (or zonal) distribution of engineering properties through the lime stabilised specimens.

5.7.4 SPECIMEN STRENGTH HETEROGENEITY

The use of clay clods result in differences in the engineering properties throughout specimens due to two processes; inter-clod porosity and then the progression of the lime stabilisation reactions at different rates in clay clods of different sizes. The evidence of this from the papers is first reviewed and then further interpretation added to link observations across the separate papers.

5.7.4.1 Inter-clod porosity

Evidence that inter-clod porosity acts as a weakness which influenced the failure planes of triaxial tests was first identified in the 32 day cures and is reported in Paper 2 (sections 3.3 and 4). Results from Paper 3 (section 5.2) confirmed that this weakness remained for 194 day cures, further noting that the inter-clod pore space would act as permeable conduit for soaking water (see Section 5.7.5 on durability).

5.7.4.2 Heterogeneous clod strength

The development of heterogeneous engineering properties across different clod sizes, was first discussed in terms of the sequence of reactions instigated by the slaking of quicklime powder to produce ‘mellowed fines’ i.e. fine clods pulverised from the periphery of larger clods (Paper 3; section 5.3). This process was clearly shown with interpretive diagrams in Paper 1
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Figure 5.10 Sequence showing how quicklime slaking increases pulverisation to produce ‘mellowed fines’ (image from Paper 1).

(a) Upon quicklime addition

(b) Mellowing - dehydration influence on ion migration

(c) Mellowing - physical effects

(d) Remixing - fine particle removal

(section 3.4) which notes that the removed fines would have a disproportionately greater amount of lime compared to the residual clod core (see Figure 5.10). Furthermore, this sequence provides a detailed explanation of why mellowing periods applied during site applications of SLS will improve pulverisation (as discussed in section 1.3.3). The evidence that lime is concentrated within these fines is in Paper 3 (section 4.2) which showed calcium contents in the finest clods of up to 7.8%, and 14-20mm clods with up to 5.2% calcium. The influence that mellowed fines had upon the specimen properties is explained Paper 3 (section 5.3) where it was proposed that the quicklime slaking process (heat and cation exchange) would cause the mellowed fines to become brittle and of relatively high strength during the mellowing period. Therefore, upon compaction the higher strength and lesser ductility of the fines would resist remoulding, unlike the larger clods, which were less influenced by the quicklime (but still coated with lime slurry during the second mix). It was proposed that this would result in a
Figure 5.11 Illustration of ‘mellowed fines’ increasing inter-clod porosity following sample compaction
(image from Paper 3)

localised increase in the inter-clod porosity weakness around these fines (Figure 5.11). This interpretation also met with the observations made during CBR specimen dissection (see section 4.1 in Paper 3), where it was noted the sample parted very easily around locations adjacent to the fines.

With regard to the rate of strength gain with long term curing, the study findings have suggested that each clod has its own internal ‘system’ bound by the clod surface (see Section 5.7.3) Considering also that an ion migration dependent reaction mechanism is proposed in Section 5.7.2, the ion gradient (essentially a function of the quantity of lime distributed around each clod and the volume that migrating Ca\(^{2+}\) and hydroxyl groups must then cover) is a key factor in determining the rate of strength gain for each individual clod. Therefore, as suggested by Petry and Wohlgemuth (1988); Bozbey and Garaisayev (2010), a greater degree of pulverisation would indeed play an important role in the efficiency of this process as the smaller clods would receive higher concentrations of lime. These differences in ion gradients would result in heterogeneous strength, whether this is caused by local variations in distribution of
lime over the clods (an unavoidable aspect of any mixing regime involving such clods; even with high efficiency site plant) or by different clod sizes.

5.7.4.3 Zonal strength with curing

The observations of inter-clod porosity and heterogeneous clod strength identified across Papers 2 and 3 may be developed into a sequence of how zonal strength within CBR and triaxial specimens evolved with curing (Figure 5.12 and 5.13). It was noted in section 3.3.2 of Paper 1 that by 32 days cure, the strength of some individual clay clods was considerably greater than other clods. This caused the stronger clods to appear as ‘nodules’ within a ‘matrix’ of softer, apparently more plastic clay material. It was estimated that these strong clods comprised around 30% of the total volume of a CBR mould and, in conjunction with the weakness from the inter-clod porosity, this caused the tested specimens to exhibit zonal strength (Figure 5.12b). By 194 days, the strength of all clods had increased substantially and the individual clods appeared to interlock, although locally the influence of fines would reduce the degree of interlocking (as per Figure 5.11). Thereby as all clay clods were relatively strong by 194 days, the air voids between clay clods would comprise significant weaknesses; especially at lower confining stress. The influence of this zonal strength was apparent when observing the morphology of triaxial failure planes which appeared to link the weak points around stronger locations, whether these were within or around the clods (Figure 5.13). This zonal strength, is suggested as the cause of the inter-specimen variability in triaxial tests, which was noted to cause some specimens to deviate from the general trend of an increase to the peak failure stress with increasing confining stress (Figure 5.5 and Figure 5.6)
Enhancing the understanding of lime stabilisation processes

Figure 5.12 Sketch indicating how zonal strength in a CBR specimen develops with curing. a, After 8 days clay clods are still relatively plastic. b, With 32 days curing ca. 30% of clods have increased notably in strength to appear brittle / friable, compared with the more plastic clod ‘matrix’. c, By 194 days strength gain in all clods is substantial and these strong clods interlock. Inter-clod porosity is visible as white areas between the clods. Mellowed fines are omitted for clarity.

Figure 5.13 Sketch indicating how triaxial failure planes may link weak zones across the test specimen.

With shorter cure periods clods are relatively weak (a) whereas with prolonged curing the failure plane links the weaker pore space around the relatively strong clay clods (b and c).
5.7.4.3.1 Validity of 38mm Triaxial Tests

To limit the influence of specimen structure from particle size effects, it is usually recommended that the maximum particle size used to form each triaxial specimen is less than 5 times the overall specimen diameter. Thereby, the value of 38mm diameter triaxial tests based on samples formed from individual clods up to 20mm may be questioned. However, the key point to this study is that the strength results were influenced by the structure and this helps confirm a lack of bonding between clods. If the addition of lime to the surface did not cause the discussed physico-chemical boundary at this point, then the individual clods should have been cemented together by a connected porewater and sample structure from inter-clod porosity would not cause the observed reductions in strength, nor pathway for soaking water (section 5.7.5). It is likely that a larger scale test (e.g. 100mm diameter triaxial sample) would more closely represent the site scale strength and experience less inter-specimen variability. Thus, while the 38mm triaxial tests likely underestimate the en-masse strength, they do evidence the presence and influence of inter-clod porosity and are valid from this perspective.

5.7.4.4 Mellowing and air voids

With the basis for inter-clod porosity and the heterogeneous development of clod strength established by the nucleation trial findings. Paper 1 (sections 3.3.1 to 3.3.3) used literature references to expand on these topics explaining how clod strength at the point of compaction is a key issue for the durability of SLS materials. This point is made with specific reference to the potentially detrimental influence that quicklime powder use with longer mellowing periods (i.e the 24 to 72 hours required by HA74; Highways Agency 2007) may impart. Based upon this, a specific conclusion of Paper 1 (bullet points 3 and 4 in Section 5.1) is that caution should be used when using longer mellowing periods as a reduced durability to soaking water ingress may result.
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5.7.5 Durability

Immersion within soaking water for 28 days caused a 20% reduction in the CBR value from 32 and 194 day cures (Table 5.9) and the manner with which soaking affected the specimens is considered. Paper 3, section 4.2 compared the water content of each gradation of clod size from the 194 day cure soaked and unsoaked CBR specimens (Figure 5.14). The nucleation centre specimens show very similar values of water content and serve as duplicate evidence of the following observations noted from the lime only specimens (Figure 5.15)

The precipitation of C-S-H within the inter-aggregate porespace (Section 5.7.2) would reduce permeability through pore blocking (Wild et al, 1987). Furthermore, it was identified from the soaked specimens that there was an increasing difference in water content with decreasing clod size. This suggests that water access through the specimens was via the inter-clod porosity and the higher surface area of the smaller clods causes the water content of these to be higher after 28 days soaking. This does suggest that after 28 days soaking, the water content change had not reached a steady state. It would be logical to assume that if soaking had continued until a steady state was reached, the water content of all clod sizes would have increased to similar values. Under such a scenario further strength reduction and specimen swell may have occurred.

Comparing calcium contents across the range of clod sizes for the soaked and unsoaked specimens from the 194 say cures identified that the soaking process did not leach a significant amount of calcium from the specimens (Paper 3, section 4.2). This suggests that the C-S-H gel formed was relatively durable to soaking and that the 20% reduction in CBR value was associated with another process e.g. softening of the residual clay minerals or loss of any negative pore water pressures.
**Research Undertaken: Nucleation Trial**

Figure 5.14 Water contents for each clod gradation from 194_CBR_L

![Bar chart showing water contents for each clod gradation from 194_CBR_L](#)

**Figure 5.15 Water contents for each clod gradation from 194_CBR_Nuc**

![Bar chart showing water contents for each clod gradation from 194_CBR_Nuc](#)
5.8 SUPPORTING EVIDENCE

This chapter is used to report the evidence that supports the key findings of the nucleation trial, but due to either the scope of Papers 2 and 3, or timing of the work were either not reported, or were only partially reported in the publications. The sub-sections presented here are cross referenced in the text of section 5.7.

5.8.1 SEM ANALYSIS

Section 4.3.3 of Paper 3 reported that SEM-EDX on clay clods from 194 day cures was used to identify the presence of C-S-H within micro-concretions up to 100μm in size and at locations within 1mm from the edge of the clay clods. The discussion in section 5.4.1 of Paper 3 then proposed that the separate diffusion of Ca$^{2+}$ and hydroxyl groups also caused the formation of C-S-H deep within the clay clods, although it was suggested the C-S-H was sub-microscopic and finely disseminated in the pore space around aggregations of clay particles. The Ca/Si ratios were used as part of the evidence for the sub microscopic C-S-H, however the scope of Paper 3 prevented a deeper explanation of the technique used. This section provides this further detail (Sections 5.8.1.1 and 5.8.1.2) to more clearly evidence how it was concluded that these Ca/Si ratios support the proposal of sub-microscopic C-S-H. Furthermore section 5.8.1.3 shows how the identification of limestone particles help to evidence that the clay clods were recovered wholly intact from the CBR specimens (as described in section 5.7.3).

It was suggested in section 5.7.1 that the novel method used to produce cross sections through the clay clods analysed in this study was the reason that regions of C-S-H up to 100μm across were identified. The preparation and analyses of samples for the SEM-EDX analysis of hydrous phases is not straightforward and requires an understanding of how atomic ratios are used. The details of the method followed, including annotated photos of the prepared sections
which show the approximate location of regions where the data points discussed in section 5.8.1.1 and 5.8.1.2 were obtained, are shown in appendix D.

5.8.1.1 C-S-H Micro-concretions

To discuss the C-S-H micro-concretions it is first necessary to explain how back scattered electron (BSE) imaging can be used to visually identify them. In BSE mode, the brightness of features in the grey-scale image is proportional to the mean atomic number of the feature. Features with higher atomic numbers appear brighter. Voids, infilled with resin, appear black. This high contrast makes it straightforward to identify porosity in such an image.

Porosity, visible at the micron scale, was present in zones throughout all peripheral and core regions of the samples. The morphology suggests that most of this void space was formed as a consequence of sample shrinkage during the drying stage of polished specimen preparation. Throughout the samples these shrinkage cracks are typically elongate (several millimetres long and up to 50µm wide), curvilinear and regularly bifurcate or undergo a sharp change in angle; presumably due to differential shrinkage of the clay mineral rich matrix (Figure 5.16). In the peripheral regions of the clay clods, a change to this dominant pore space morphology was noted as the C-S-H micro-concretions were surrounded by annular pores of some 3 to 10 microns wide. These annular cracks provide an indication that the C-S-H shrunk more than the surrounding material and helped to identify the C-S-H accumulations when viewing a field of view of larger scale (Figure 5.16). This helped to delineate areas with the C-S-H micro-concretions for EDX analysis which would allow the conclusion that no C-S-H micro-concretions were observed beyond 1mm into the clay clods.
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Figure 5.16 SEM BSE image illustrating features from area P3. Voids (infilled with resin) appear black. Example ‘elongate’ shrinkage cracks are labelled. Areas between the nodule edge and dashed line comprise regions where annular porosity around accumulations of C-S-H are apparent.

5.8.1.2 Sub-microscopic C-S-H

When comparing the Ca/Si ratio of EDX analyses of C-S-H with those from matrix areas both near the clay clod periphery (see Figure 4 and Table 2 in Paper 2) and those from the clod core, a pattern became clear. Analyses from matrix regions were characterised by a Ca/Si ratio that falls somewhere between the values for the C-S-H micro-concretions and quartz (quartz having zero calcium). Plotting the atomic percentage of calcium (Ca%) versus the Ca/Si ratio for all EDX analyses (excluding those of quartz) that were carried out for this research suggests these values are related (Co-efficient of determination \( R^2 = 0.976 \); Figure 5.17)
Figure 5.17 Atomic calcium % versus Ca/Si ratio for all EDX analyses, excluding those of quartz, from areas P1-P3 and C1-C3. The dashed lines segregate two groups of data; 1 – analyses from C-S-H accumulations; and 2 – analyses from all other locations. Both groups are closely clustered around a common trendline of $R^2 = 0.976$. Some outliers occur (9 from 94 measurements) and these are thought to represent unaltered minerals or perhaps different reaction products. These outliers are outside the dashed lines and omitted from the trendline.

In this plot, values from the higher end coincide with spectra taken within C-S-H micro-concretions, whereas values from the lower end are from all other matrix locations. Both sets of values can be characterised by the same line and this suggests that, regardless of the total calcium %, the proportion of calcium matches the silica at a relatively constant ratio. The trendline therefore suggests that the composition of the reaction product is similar, i.e. C-S-H, throughout the clay clods.
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5.8.1.3  Limestone at nodule edge

For the samples with nucleation centres, the limestone powder was useful in providing further confirmation that the 194 day specimens did separate as intact clods. During mixing, some of the coarser particles of limestone did not dissolve into the porewater and a white coating remained as a partial covering to clod surfaces throughout mixing and after curing. The limestone particles were identified during the SEM analysis to a maximum of 400µm from the edge of the nodule (Figure 5.18) and not at any field of view deeper within the clods.
5.8.2 XRD Analysis

Paper 3 (section 4.3.2) noted that the XRD analysis provided limited information about the reaction products although examples of the XRD traces were not included. Figure 5.19 compares changes to the XRD trace for the untreated clay from the 32_L and 194_L cures. Furthermore, a comparison of the XRD traces from the core and peripheral regions of 14-20mm nodules from 194_L and 194_Nuc are provided (Figure 5.20).

![XRD traces for Cauldon clay and lime stabilised batches (32 and 194d cure). Vertical lines indicate the location of new peaks (associated with the noted minerals) present in some cured samples.](image)

Figure 5.19 XRD traces for Cauldon clay and lime stabilised batches (32 and 194d cure). Vertical lines indicate the location of new peaks (associated with the noted minerals) present in some cured samples.
Figure 5.20 XRD traces for Cauldon clay and regions of 14-20mm clay clods of 194d cure (Periphery and core). Vertical lines indicate the location of new peaks (associated with the noted minerals) present in some cured samples.

The data from the 32_L samples data show that the only new peaks identified were associated with portlandite (i.e. the lime added during mixing) and calcite (Figure 5.19). As calcite was not added to the lime only specimens, it is likely that some of the portlandite was carbonated by exposure to air during preparation (drying/grinding) of the specimens for XRD analysis. The samples from peripheral regions of the 194 day cures also show strong reflections for the
minerals that were directly added i.e. portlandite for lime only and both portlandite and calcite for the nucleation samples (Figure 5.20). The core locations from both lime only and nucleation samples show an absence of any new reflections. This supports conclusions that:

1. The core sub-samples were from regions deep with clay clods and that they were not in direct contact with lime / limestone powder during mixing;

2. Lime migrates as separate ion groups of Ca\(^{2+}\) and OH\(^{-}\) and not as molecules of Ca(OH)\(_2\). This argument is further strengthened when considered with the results showing a substantial increase to Ca\(^{2+}\) and pH deep into the 14-20mm clay clods (see Figure 3 in Paper 2).

3. The C-S-H formed in this study was poorly crystalline and not detectable by XRD. This is a common challenge as when XRD analyses is undertaken on pure C-S-H specimens limited diffraction peaks typically result (Viehland et al., 1996).

5.8.3 **THERMAL ANALYSIS**

Subsequent to the publication of Paper 2, TGA was undertaken on the sub samples from core and peripheral regions of the 14-20mm clay clods from 194_L_CBR_S specimens (sub sampling process is described in section 2.4 of Paper 2). Detail on how TGA was used in this research is given in section 6.4.2 and Table 6.10.

Comparison of derivative plots from the untreated Cauldon clay and 194 day cures reveal that there are no clear new peaks in the lime treated specimens (Figure 5.21). While it may be argued that when compared with untreated clay, both 194_L specimens show a minor reduction to the height of the peaks centred 70-75°C and 275°C, there cannot be strong confidence that this is influenced by lime-clay reactions. Variations of this magnitude may be sourced from the host clay soil. As portlandite was noted in the XRD trace for the 194_L
periphery sample, the absence of any peak around 450°C is surprising (Table 6.10). This perhaps highlights the difficulty of using this technique when the weight loss from minerals in the host clay and expected new minerals occur at similar temperatures. In this regard the Cauldon clay was particularly ‘noisy’ in the region where Portlandite (approx. 450°C) and C-S-H (65-160°C) should reflect (Table 6.10). This difficulty may be further compounded when considering the quantity of new formed minerals were suspected to be of a very low percentage compared with those in the host clay soil. Therefore, the TGA analysis neither confirms nor detracts from the mechanism of C-S-H formation discussed to this point.

![TGA derivative curves (DTA) for Cauldon clay and select regions of 14-20mm clods from 194_L_CBR_S specimens. Note that for sample “194_L 14-20mm periphery” no data was recorded between 404°C - 417°C, which was due to interference from an electrical device.](image)

Figure 5.21 TGA derivative curves (DTA) for Cauldon clay and select regions of 14-20mm clods from 194_L_CBR_S specimens. Note that for sample “194_L 14-20mm periphery” no data was recorded between 404°C - 417°C, which was due to interference from an electrical device.
5.8.4 **Summary**

The key findings of the nucleation trial have been that specimens of a lime treated clay soil prepared using a site realistic method continue to increase in strength over 8, 32 and 194 day cure periods. The sample strength has been correlated with:

- Micro-chemical data to propose how ion migration permits diffuse cementation through large clods;

- Macro-structure to demonstrate how large scale structure reduces specimen strength and increases susceptibility to strength reduction from immersion in water.

It was noted that specimen preparation and particularly the use of longer mellowing periods has the potential to significantly increase the degree of inter-clod porosity and worsen specimen durability to water ingress. This observation was included within Paper 1 and was taken forward for close consideration in establishment of the sulphate swelling trial and this is discussed in Section 6.1.

There was little evidence to suggest that addition of nucleation centres had increased the strength of these specimens, although the primary reason for this appears to be the inter-clod porosity between clods. Therefore, the compacted and cured lime treated specimens, both with and without nucleation centres, appear to behave as a discontinuum of individual clods. This is rather than the continuum of clods bound together by pozzolanic reaction as was the expectation at trial outset. However, conditioning samples to a water content wet of the OWC appears to have facilitated the development of diffuse cementation through larger clods and this observation was also considered when establishing the basis of the sulphate swell trial (Section 6.1)
6 SULPHATE SWELL TRIAL

This chapter sets out how a laboratory trial into sulphate swelling investigated methods to reduce the risk of sulphate swell under site relevant conditions. Due to the end of this trial running close to the end of the EngD study period, there are yet to be any publications relating to the findings of the sulphate swell trial. Therefore, unlike the thesis text discussing key findings from Paper 2 and 3 (Chapter 5) cross-referencing to appended publications is a minor aspect of this chapter. While the work follows on from the nucleation trial and there is some interpretation of the discussions from Paper 1, this Chapter is structured (sub-sections as below) to report and discuss findings in a relatively standalone manner.

Sub-section 6.1 reviews the literature considered upon formulation of the trial approach and discusses these in relation to relevant findings from the nucleation trial.

Sub-section 6.2 details the methodology followed to assess how variations in the binders used and mellowing periods influence the degree of swell after prolonged soaking. The use of a novel test to determine swelling under conditions more relevant to those encountered on site is explained and reasoned.

The results are presented and analysed in sub-sections 6.3 and 6.4. Sub-section 6.3 focuses on physical measurements (e.g. swelling), then sub section 6.4 comprises an assessment of mineralogical changes.

In sub-section 6.5 the trial results are discussed and compared with the theoretical basis established at trial outset. Conclusions relevant to the industry sponsors are then made.
6.1 LITERATURE / NUCLEATION TRIAL CONSIDERATIONS

Paper 1; section 4.3 discussed key literature on how sulphate swell develops in lime stabilised soils. This included a summary of three areas where the literature had made suggestions on how sulphate swell (primarily from ettringite growth; see Section 4.1.2) may be reduced or controlled. It was concluded that the main challenge for industry was a lack of clarity on how these strategies may be incorporated into a site working method using large clods. Furthermore, in context with the nucleation trial findings some of these strategies appeared to conflict with each other. To clarify the key areas considered when finalising the trial methodology the sulphate swell control strategies from literature, along with the potential points of conflict, are summarised in sections 6.1.1 to 6.1.3. Then the influence of the site environment is considered in section 6.1.4

6.1.1 LIMITING REACTANTS

6.1.1.1 Underlying Principal

The principal underlying this sulphate swell control measure would be to limit the supply of one or more of the reactants required in the molar concentrations noted in equations 4.2 and 4.3, thus reducing growth. As the Al₂O₃ (from clay minerals) and SO₄²⁻ (e.g. from gypsum or pyrite) are natural components to the clay soil, other than avoiding clay rich and/or high TPS soils, there is limited potential to isolate these reactants. Ca²⁺ may be present naturally (e.g. from calcite or gypsum), however, the added lime will typically be the major source. While it may be counter-intuitive to the principal of soil stabilisation, to lessen sulphate swell risk it may be possible to add less lime, especially if used in conjunction with a secondary binder (e.g. GGBS). Limiting the supply of H₂O into a compacted system may be the most viable mechanism of limiting ettringite formation. This would meet well with ISC’s observation
where improved site drainage was noted to arrest an ongoing swell issue (section 1.6.3). However, as groundwater regimes may change unexpectedly or drainage systems develop faults, it is principally difficult to ensure a near surface environment will not become immersed.

### 6.1.1.2 Nucleation Trial Lessons.

The inter-clod porosity and potential role as a conduit for soaking water through clods (see section 5.7.5) is of key interest. Therefore, thoughts on how preparation can either minimise water ingress through this route are key. As the use of quicklime appeared to increase the strength of the mellowed fines before compaction, and subsequent degree of inter-clod porosity around these fines (Figure 5.11), it may be preferred to instead use hydrated lime.

When considering the permeability of the individual clods, it was striking how resilient the larger clods of the 194_CBR_S were to 28 days immersion in water (see Section 5.7.5). As noted in section 5.7.5, while aggregation of clay minerals may increase permeability via inter-aggregate pore space in the short term, the long term pozzolanic reactions then precipitate in this region to reduce permeability through the ‘pore blocking’ process described by Wild et al (1987). Therefore, a significant cure period before immersion in water may well allow a degree of pore-blocking to develop.

### 6.1.2 CONTROLLED ETTRINGITE GROWTH

#### 6.1.2.1 Underlying Principle

Ettringite is only expansive if the water is sourced from outside the immediate system i.e. post compaction (Little et al., 2010). Furthermore, where all reactants are immediately available (i.e. intimately mixed and in the correct molar concentrations), ettringite will form and reach a steady state within 150 hours (Little et al., 2005). However, the heterogeneous distribution of reactants, in combination with the low solubility of sulphate will further slow ettringite
precipitation in a site application. If diffusion of reactants is encouraged through addition of sufficient water at the mixing stage (3-5% greater than the OWC) and this is coupled with an extended mellowing period (e.g. 3-7 days), this promotes the widespread nucleation and some early ettringite growth before final compaction takes place (Little et al., 2010; Petry and Little, 2002). This strategy will limit expansion by encouraging initial ettringite growth before compaction, and then facilitating any subsequent expansive ettringite growth (as sulphate solubility allows) over a greater area. The latter would promote the wider distribution of smaller ettringite crystals, so there is increased potential for this volume change to be accommodated by the inter-clod pore space; thus avoiding expansion hotspots (Little et al., 2010).

**6.1.2.2 Nucleation Trial Lessons**

The concept of using extra water to encourage early diffusion and ettringite nucleation throughout clods meets well with the findings of the nucleation trial and from this perspective has potential as a method. However, the use of extended mellowing periods will allow time for the early lime clay reactions to progress, which may well lead to individual clay clods developing significant strength to resist compaction and increase the degree of inter-clod porosity; especially if quicklime is used (see Section 5.7.4.2). As crystalline ettringite is considered to add substantial strength to an SLS soil, this effect may be worsened if significant ettringite growth does occur during mellowing. However, as Little et al.’s (2005 & 2010) work was based upon regions of the United States where hydrated lime is typically used, it may be that the absence of heat (from quicklime slaking) lessons the rate of early lime reactions. Therefore, with hydrated lime and the frequent addition of mixing water through mellowing, it may prove that the water content can match increases to the plastic limit, thus allowing the clods to remain ductile during compaction; lessening the degree of inter-clod porosity.
6.1.3 **Chemical Balance / Timing**

6.1.3.1 Underlying Principle

Increasing silica activity in a clay soil that has a chemistry that would normally form ettringite could instead lead to the formation of a volumetrically stable mineral, e.g. the calcium alumino sulfo silicate – prehnite (Little et al., 2005). Thus binder mix designs including products with rapidly available silica such as GGBS have been linked with the prevention of sulphate heave (Higgins, 2005). As an alternative hypothesis to how GGBS restricts sulphate heave, Wild, et al., (1996) suggested that the GGBS particles, which also contain aluminium, provide an alternative surface for ettringite nucleation. They proposed that ettringite growth extending from the surfaces of GGBS particles was non-expansive, whereas if ettringite growth nucleated from the edge site of a clay mineral this was associated with expansion (Wild et al., 1996). It may be that the non-expansive nature of ettringite growth in this case was a function of the rapid supply of aluminate (rapidly dissolved from a relatively amorphous GGBS source) to combine with the other immediately available reactants thus permitting the rapid formation of ettringite in the controlled manner described in section 6.1.2.1.

6.1.3.2 Nucleation Trial Lessons

The nucleation trial proved that Ca(OH)₂ does partially dissociate into Ca²⁺ ions and hydroxyl groups which diffuse and through and react deep into the clay clods. However, it is not clear whether GGBS would be able to migrate in the same manner, or whether the high reactivity of GGBS with lime would cause these effects to be limited to the surface of the clay clod. Wilkinson et al. (2010) used ESEM images to illustrate how clay slurries treated with lime and GGBS experienced the substantial growth of pozzolanic reaction products (primarily C-S-H) from the surface of clay particle aggregations and described this as a through solution process.
The rate of C-S-H growth in the lime / GGBS specimens was much greater than the lime only equivalents and was directly correlated with improvements to engineering properties, with the alkaline hydrolysis of the GGBS considered as the rate limiting factor (Wilkinson et al., 2010). This suggests that any diffusion of dissolving GGBS particles would be relatively limited and the resultant reaction products would be closely confined to the clod surface. Thereby, the swell suppression from this process would similarly be limited to the clod surface. However, it is worth noting that the rapid growth of reaction products at this location may well reduce the permeability of the clods by ‘blinding’ the surface. Thus, addition of GGBS may also / instead reduce sulphate swell by limiting water access and ettringite growth through the process described in 6.1.1.1)

6.1.4 ENVIRONMENTAL CHANGES

A potential issue with using methods that encourage ettringite formation before compaction is that a reduction to temperature after compaction, (e.g. onset of winter) may change the stability of ettringite (see 1.6.3.1). Reduced temperature may cause ettringite growth to increase (see Section 4.1.2) and further swell may result due to this. Furthermore at temperatures <15°C and with a source of carbonate / silicate, ettringite may convert to thaumasite and trigger further swell and strength loss. Snedker (1996) noted the increase in swell from such temperature reductions during on site soaked CBR testing on the M40 failure. A loss of power to the soaking tank heater caused the water temperature to drop below 20°C for several days before the problem was corrected. Snedker (1996) describes how a sudden increase in swell readings occurred as the temperature fell below 15°C, returning to the lesser swell rate as the water temperature recovered to 20°C. This caused Snedker (1996) to question the validity of standard tests which specify a 20°C temperature and to recommend further investigation into this apparent swell increase at lower temperatures. Notwithstanding, the two British Standard swell
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tests, i.e. the soaked CBR and accelerated Swell test (see Section 6.1.4.1) require soaking water temperatures of 20°C and 40°C respectively (BSI 2004a; BSI 2004b).

6.1.4.1 Standard Swell Tests

As discussed in Section 1.6.1.2, subsequent to M40 failure, the soaked CBR test (as described by BSI, 2004a) has been the routine means of assessing the potential for sulphate swell risk in the UK. The mould, within which the material is compacted, confines the CBR specimen at all sides except the top where a swell plate is used to determine the linear expansion of the plate (measured in millimetres; mm) upon immersion in 20°C water.

In continental Europe it is more usual for the accelerated swell test (as described by BSI, 2004b) to be used for determinations of sulphate swell risk. The 2004 provision of a British Standard provided the option to use this method for assessing sulphate swell in the UK. The accelerated swell test is undertaken on 50mm x 50mm prisms of axially compressed material. Prior to compression, the soil is passed through a 6.3mm sieve and treated with the binder, mixing water and mellowing periods as required. The formed sample is measured to determine the initial volume, sealed and cured in air at 20°C for 72 hours. The sample is then wrapped in a geofabric which is secured with elastic belts and immersed in 40°C water for 168 hours. Upon completion of soaking the specimen volume is determined and the percentage of volume change calculated. For a pass/fail criteria, BSI (2006; p12) stipulates “where the volumetric swelling is greater than 5 % but does not exceed 10 %, the use of the mixture is generally not possible; however a complementary study can be made according to experience at the place of use”.

Thus the failure point is typically taken as a 5% volume increase.

As the two standard methods have some significant differences, this had led to two studies considering whether one is more suitable than the other in the identification of sulphate swell risk. Notman (2011) suggests the CBR test is less stringent than the accelerated swell test
and that the pass / fail criteria of the former is more likely to identify material which may undergo a high degree of swell in the field. Notman (2011) suggests the steel CBR mould may comprise an artificial boundary around the compacted sample which significantly restricts soaking water ingress (i.e. through small holes in the top and bottom soaking plates only). Furthermore, friction/adhesion forces between the CBR mould and sides of the soil were considered of such magnitude that they would suppress expansion from increased swell pressures, thus avoiding detection by swell plate readings. The accelerated swell test does not have this degree of confinement and Notman (2011) proposed the very test set up of the soaked CBR would limit its potential to identify sulphate swell relevant to site conditions. While this may provide some support to an argument in favour of an unconfined specimen, it should be noted that the absence of any confining pressure to the specimen may also be unrealistic. If unconfined, expansion will widen porosity between clay clods and allow a higher degree of permeability than would occur within a pavement foundation. This may exaggerate softening and swelling of the clay minerals within the lime treated specimens, not necessarily from sulphate swell reactions. This may explain why Buttress et al. (2014) noted that a lime stabilised kaolinite soil of minimal TPS exceeded the fail criteria of the accelerated swell test.

Higgins et al. (2013) also compared the soaked CBR and accelerated swell tests and concluded that both tests provided very similar results. It was suggested that both tests were successful in the identification of swell potential when the sulphur species was sulphate, but neither test identified the swell potential of a pyritic clay of high TPS. However, it was concluded that the quantity of lime binder used, which was less than the ICL value, may not have been high enough to fully stabilise this pyritic clay (Higgins et al., 2013).

Other potential issues with the standard swell test methods is that both involve full immersion of the prepared specimen after very short unsoaked cure periods (i.e. 72 hours cure @ 20°C). This allows little time for the development of any initial pozzolanic reactions (curing
conditions are discussed further in section 6.2.4.2). Both tests involve full immersion in water which avoids a humid / oxidising atmosphere and this may prove significant for clay soils with a substantial oxidisable sulphur content (e.g. pyrite). While extended mellowing periods with frequent mixing / water addition may encourage pyrite oxidation before compaction (Floyd et al., 2003), short mellowing periods may include a residual risk of pyrite oxidation and further sulphate swell after compaction (This is discussed further in section 4.3.4 in Paper 1). Therefore, where pyritic soils are treated with SLS, a swell test that includes a phase with an oxidising and humid atmosphere may be important.

6.2 SULPHATE SWELL TRIAL METHODOLOGY

6.2.1 NOVEL SWELL TEST

Due to the limitations in the existing test methods (Section 6.1.4.1) it was considered important to first devise a Novel Swell Test (NST) which would detect sulphate swell mechanisms operative in the site scenario. The NST is briefly described with reference to a general arrangement drawing in section 6.2.1.1 and Appendix E includes full details of the test set up. The key features including aspects specifically intended to address the limitations associated with the existing sulphate swell tests (Section 6.1.4.1) are then explained in section 6.2.1.2

6.2.1.1 General Arrangement

The NST (general arrangement in Figure 6.1) examines the degree of swell from a cylindrical specimen (approximately 102mm diameter and 116mm length). This requires that in advance of the test, the specimen is formed in a split proctor mould, demoulded, sealed and cured at 20°C for the required period. After cure, the specimen is covered in a non-woven needle punched geo-fabric and placed within a CBR mould set up with soaking plates / collar. Thereafter, the space between the specimen, CBR mould and swell plate is filled with single
sized (1-2mm) Leighton buzzard sand (Figure 6.1). The reason for the geo-fabric was to separate the sand, preventing contamination of the specimen causing difficulty with executing the post swell tests. The NST arrangement is then placed in soaking water and the sample swell response determined from swell plate readings. For the purpose of this study, the specimens were to remain in the soaking water until sample swell reached a steady state i.e., when the difference in swell plate readings is less than 0.02mm over 3 consecutive days. The actual volume change in the specimen would be determined by comparing the initial sample volume (as measured with Vernier calipers) with the final volume (determined using the volume on immersion test, BSI 1990a) to calculate the percentage of volume change.
6.2.1.2 Key Features

A significant feature of the test is that highly permeable Leighton Buzzard sand allows soaking water to rapidly access all sides of the specimen. The sand would also provide radial confinement to the swelling sample, an important aspect as it was intended for swell plate readings to provide the primary indication of the rate of volume change during specimen soaking. This assumption is valid if the specimen is confined except for the top surface, and compaction of the single sized, high sphericity sand grains (compaction process described in Appendix C) were successful in minimising air voids. Accordingly, increases in the one dimensional swell readings should provide a close indication of volume change in the specimen and any processes which suddenly change the swell rate.

A further reason for including the Leighton Buzzard sand layer was that it has possible use in providing an oxidising atmosphere around the sample; potentially important for sulphate swell in pyritic clays or thaumasite formation (see Sections 6.1.5.1 and 6.1.1). An oxidising environment could be encouraged by aerating the soaking water, or by reducing the water level in the soaking tank to below the base of the CBR mould. The latter should allow water to drain out the sand layer and leave the specimen sides adjacent to sand with voids filled with air of a high relative humidity. However, to limit experimental variables this step was not actually included in this study and is instead a possible future adaptation.

Another unique aspect of the NST is that it includes a reduction in the temperature of the soaking water to investigate whether this triggers a change in the swell rate. For this comparison two NST’s were required for each batch. The first is used as a control and soaked in a 20°C tank for the full duration of the test (herein referred to as NST 20°C). The other NST specimen is soaked in its own bucket where the water is maintained at 20°C for the first 35 days (cure periods explained in Section 6.2.4.2), before transferal of the bucket to an environmental
chamber held at 8 °C for the remainder of the test duration (herein referred to as NST 8°C). Section 6.2.1.4 explains why 8 °C was used for the reduced temperature.

### 6.2.1.3 Soaked CBR comparison

To assess the NST against results from an existing and accepted test method, it was planned that in addition to a NST 20°C and 8°C specimen a standard CBR specimen would be prepared with each batch. The CBR would be cured and soaked in the same manner as the NST 20°C test.

While it would have been ideal to also include an accelerated swell test with each batch, the extra soil and time required to achieve this was a limiting factor. This required that a choice between the standard swell tests be made and the soaked CBR was selected for several reasons including continuity in approach from the nucleation trial. The study focus on using large clay clods as part of the preparation method (see Section 3.2) conflicting with the requirement for accelerated swell test specimens to be formed from <6.3mm clods (see Section 6.1.4.1) was the most significant reason.

### 6.2.1.4 NST Practicalities

As it was intended to create a NST which the sponsor companies could execute at their own laboratory facility, the method used elements that could be reproduced in this environment. ISC already own a significant amount of soaked CBR equipment and this is one reason why CBR equipment was adapted to ‘host’ the swell test. Similarly, Leighton Buzzard sand is widely available in the UK and often stocked in geotechnical test laboratories e.g. for use in sand replacement tests.

The reason that 8°C was selected for the reduced temperature is that consultation with a field lab manager suggested this is a realistic temperature that a site unit (e.g. shipping container
style) could be maintained at all year round using widely available refrigeration equipment. Temperatures lower than this may require specialist and more expensive apparatus. The 8 °C temperature was considered adequate as it is within the thaumasite stability range (section 4.1.2), and while not the worst case, is within the temperature range foreseeable for the near surface environment during a UK winter.

It was anticipated that the NST would realise the full degree of ettringite growth / sulphate swell in a shorter timeframe than the soaked CBR test. Where sulphate swell is extensive, the soaked CBR test can take months for the swell to reach a steady state and such a long delay with a pre-contract suitability test may prevent the true risk being understood at point of contract tender. As the NST features a soil specimen which is approximately 2.3 time smaller in volume than the soaked CBR and that water access to all sides is permitted from the test start, it was foreseen that all swelling in the NST would complete much sooner than the comparable soaked CBR. Therefore, the NST may prove a more suitable test for pre-contract testing from both a speed and suitability perspectives.

6.2.2 Preparation Method

As discussed in section 6.1.1-6.1.3, there were several suggestions from the literature on how sulphate swell may be reduced and the strategies of increased mellowing / mixing water addition (e.g. Little et al., 2010) and the role of GGBS in reducing sulphate swell (e.g. Wild et al., 1999) were directly trialled. Furthermore, a principle of preparation that sought to minimise soaking water permeability through the inter-clod porosity of the compacted / cured specimens was also followed. Based on the theory in section 6.1.1-6.1.3, this should provide the best opportunity for one of the trialled measures to limit sulphate swell (from the test clays of very high TPS; details in section 6.2.3) to an acceptable value. To minimise inter-clod porosity at compaction, hydrated lime powder (provided by Lhoist; product name ‘Proviacal H’) was used
instead of quicklime and mixing water was added to target an MCV of 10. Furthermore, to allow time for initial lime clay reactions to progress and reduce the permeability of individual clods before immersion in soaking water, an unsoaked cure period before immersion of 28 days at 20°C was used (further discussion in section 6.2.4.2).

6.2.2.1 Batches

For each of the two test clays soils used (Aylesbury and CaulGyp, see section 6.2.3) one batch was prepared using lime only, while for the second batch, the lime binder was partially replaced with GGBS (at a ratio of 3 GGBS: 1 lime). Thereafter, sub-batches from both lime and GGBS batches would be subject to different mellowing periods and mixing water addition. In total 12 different sub-batches were prepared, as summarised (along with a shorthand reference) in Table 6.1.

<table>
<thead>
<tr>
<th>Clay</th>
<th>Batch (binder type)</th>
<th>Mellowing period</th>
<th>Sub batch No.</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aylesbury Lime</td>
<td>1 Hour</td>
<td>1</td>
<td>Ay_1_L</td>
<td></td>
</tr>
<tr>
<td></td>
<td>24 Hours</td>
<td>2</td>
<td>Ay_24_L</td>
<td></td>
</tr>
<tr>
<td></td>
<td>72 hours</td>
<td>3</td>
<td>Ay_72_L</td>
<td></td>
</tr>
<tr>
<td>Aylesbury Lime + GGBS (ratio 82.25:18.75)</td>
<td>1 Hours</td>
<td>4</td>
<td>Ay_1_G</td>
<td></td>
</tr>
<tr>
<td></td>
<td>24 Hours</td>
<td>5</td>
<td>Ay_24_G</td>
<td></td>
</tr>
<tr>
<td></td>
<td>72 Hours</td>
<td>6</td>
<td>Ay_72_G</td>
<td></td>
</tr>
<tr>
<td>Cauldon clay with 5% Gypsum Lime</td>
<td>1 Hours</td>
<td>7</td>
<td>CG_1_L</td>
<td></td>
</tr>
<tr>
<td></td>
<td>24 Hours</td>
<td>8</td>
<td>CG_24_L</td>
<td></td>
</tr>
<tr>
<td></td>
<td>72 Hours</td>
<td>9</td>
<td>CG_72_L</td>
<td></td>
</tr>
<tr>
<td>Cauldon clay with 5% Gypsum Lime + GGBS (ratio 82.25:18.75)</td>
<td>1 Hours</td>
<td>10</td>
<td>CG_1_G</td>
<td></td>
</tr>
<tr>
<td></td>
<td>24 Hours</td>
<td>11</td>
<td>CG_24_G</td>
<td></td>
</tr>
<tr>
<td></td>
<td>72 Hours</td>
<td>12</td>
<td>CG_72_G</td>
<td></td>
</tr>
</tbody>
</table>

Table 6.1 Batches and sub batches prepared during the sulphate swell trial.

6.2.3 The Test Clays

As with the nucleation trial, it proved difficult to source the ideal natural clay for use in the trial. The preferred test clay would have similar properties to the ideal clay for the nucleation trial (section 5.4), however, it would also have a TPS substantially in excess of the 1%
maximum value stipulated in HA74 (The Highways Agency, 2007). Furthermore, as the most challenging material appeared to have a significant oxidisable sulphur content, e.g., the pyritic Lower Lias clay associated with the M40 failure, this type of material was specifically sought. Ultimately, the timely sourcing of a pyrite rich Lower Lias material did not transpire and the below two high TPS clays were used instead.

6.2.3.1 Aylesbury Clay (Ay)

A medium-high plasticity clay soil (Atterberg limits in Table 6.2) from the weathered Kimmeridge Clay formation was obtained from a site in Aylesbury, Oxfordshire. The soil was recovered from a single machine excavated trial pit at a depth approximately 3-3.5 metres below ground level and was described as “grey soft consistency low strength CLAY with frequent selenite (gypsum) crystals. The selenite crystals were typically 3-7mm, however, rare crystals were up to 20mm in diameter”.

<table>
<thead>
<tr>
<th>Test name</th>
<th>Ay</th>
<th>CaulGyp</th>
</tr>
</thead>
<tbody>
<tr>
<td>Total Potential Sulphate (%)</td>
<td>3.4%</td>
<td>2.5%</td>
</tr>
<tr>
<td>Total Sulphur (mg/kg)</td>
<td>11300</td>
<td>8120</td>
</tr>
<tr>
<td>Acid Soluble Sulphate (mg/kg)</td>
<td>26000</td>
<td>25000</td>
</tr>
<tr>
<td>Oxidisable Sulphate (mg/kg)</td>
<td>8000</td>
<td>640</td>
</tr>
<tr>
<td>Plastic Limit (%)</td>
<td>24</td>
<td>26</td>
</tr>
<tr>
<td>Liquid Limit (%)</td>
<td>49</td>
<td>59</td>
</tr>
<tr>
<td>Plasticity Index (%)</td>
<td>25</td>
<td>33</td>
</tr>
</tbody>
</table>

Table 6.2 Sulphate and Atterberg test results for the Ay and Cg clays. Note these tests were by a commercial laboratory (CMT KIWA in Derby, UK) and are not the author’s direct work.

The Aylesbury (Ay) clay had a TPS of 3.4% (tested sample excluded the large gypsum crystals), of which the majority was in the form of sulphate (presumably from the gypsum), although approximately 24% of the TPS was from oxidisable sulphur. Pyrite is known to occur within the Kimmeridge clay (Building Research Establishment, 2005) and this is the likely source of the oxidisable sulphate. While, pyrite was not apparent from the XRD traces (Figure
6.17), this may be due to relatively low quantity being below the limits of detection by XRD (minimum 3-5%).

Prior to treatment with binders and using the same method for the nucleation trial (as described in section 2.2.1 of Paper 2) the Ay clay was hand pulverised into clods and passed through 20mm, 14mm, 10mm and 5mm sieves. Each batch of prepared materials received the same proportion of each clod size, which is shown in Table 6.3 along with initial water contents.

<table>
<thead>
<tr>
<th>Clod size</th>
<th>Mass in each batch (%)</th>
<th>Water content (%)</th>
<th>Mass in each batch (%)</th>
<th>Water content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;5mm</td>
<td>6.4</td>
<td>34.1</td>
<td>Not tested</td>
<td>35.3</td>
</tr>
<tr>
<td>5-10mm</td>
<td>28.2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>10-14mm</td>
<td>23.4</td>
<td>32.2</td>
<td></td>
<td></td>
</tr>
<tr>
<td>14-20mm</td>
<td>42</td>
<td>32.3</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 6.3 Clod size distribution and water content, for each batch of Ay and CG clay

6.2.3.2 Cauldon Clay + 5% Gypsum (CG)

This comprised use of the high plasticity Cauldon Clay material (used in the nucleation trial), artificially spiked with 5% of gypsum powder (provided by Saint Gobain; product name ‘Superfine White’). To produce clay clods of this artificial clay in a consistent manner, the below method of reconstitution was followed.

The natural Cauldon Clay was oven dried at 100°C and then pulverised by a hammer mill into particles of <500µm. This clay powder was then extensively blended, with the later stages of blending including addition of 5% (by dry mass) of gypsum powder. Batches of the well mixed clay/gypsum powder were then combined with a set volume of water and reconstituted by a Z-blade mixer. The water addition targeted a water content of 31% (which would have been 1.2 times the plastic limit; Table 6.2), however, the average water content achieved was higher 35.3% (Table 6.3). The reconstituted Cauldon clay + 5% gypsum (CG) were stored in sealed bags for 72 hours before hand pulverisation through a 20mm sieve. Due
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to the high water content of the CG clay, the clods were soft and of significant plasticity, which made it impractical to sieve and obtain a grading.

The TPS of the CG clay was 2.5% and as the Cauldon clay was of a very low sulphur content, this was almost exclusively from the added gypsum (Table 6.2).

6.2.4 **Changes to Site Realistic Preparation Method**

The method used to inter-mix the binder / water around the clods was very similar to that used in the nucleation trial study (see Paper 2; Section 2.2.2). The primary difference was the incorporation of further mellowing periods and extra mixing water addition (Figure 6.2). The use of four batches (Table 6.1) required that the process (Figure 6.2) was completed four times and the preparation of all batches was staggered over 5 weeks.

The amount of binder added was 8% by dry mass of the untreated soil and this was added in two stages (Figure 6.2). The first stage comprised the addition of lime at a quantity to equal the ICL value; which for the Ay clay had been determined (using hydrated lime) as 4.67%. When the Cauldon clay ICL value of 3.53% (using quicklime; Table 5.5) was converted to an equivalent hydrated lime value, this was very close to the Ay ICL value, so for simplicity an ICL of 4.67% was used for both Ay and CG clays. It was hypothesised that the added lime would begin to diffuse through the clay clods, facilitating early nucleation of ettringite as mellowing progressed and further mixing water was added (Little et al, 2010; Figure 6.2). The remaining 3.33% of binder was added after the mellowing period. The second phase of lime addition, closely followed by further mixing water would then promote lime diffusion and non-expansive ettringite growth deep into the clods during the 28 days unsoaked curing phase (Wild et al., 1993; Little et al, 2010). A similar process was expected with the GGBS batches, however, it was considered likely that the rapid reactivity of GGBS would cause growth of reaction products at the periphery of clay clods, restricting the later ingress of soaking water (as
Whenever water was added during mixing it was intended to use a quantity sufficient to achieve a MCV value of 10; thus maintaining a water content several percent wet of OWC.
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throughout mellowing. Due to limited material, the MCV was not actually confirmed until immediately before specimen compaction and the intermediate assessments were based on judgement from the nucleation trial and on-site experience.

6.2.4.1 Test Specimens and Quality Control

For each of the 12 sub-batches two proctor specimens (for the NST 20°C and 8°C tests) and one CBR mould were prepared using 2.5kg (normal proctor) compaction. To confirm the water conditioning had been successful, an MCV test was undertaken immediately prior to specimen compaction. The MCV’s obtained are closely comparable confirming relative consistency in the degree of water conditioning across the sub-batches (Table 6.4).

<table>
<thead>
<tr>
<th>Sub Batch</th>
<th>MCV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ay_1_L</td>
<td>10.4</td>
</tr>
<tr>
<td>Ay_24_L</td>
<td>9.8</td>
</tr>
<tr>
<td>Ay_72_L</td>
<td>10.3</td>
</tr>
<tr>
<td>Ay_1_G</td>
<td>9.8</td>
</tr>
<tr>
<td>Ay_24_G</td>
<td>10.4</td>
</tr>
<tr>
<td>Ay_72_G</td>
<td>10.2</td>
</tr>
<tr>
<td>CG_1_L</td>
<td>9.8</td>
</tr>
<tr>
<td>CG_24_L</td>
<td>10</td>
</tr>
<tr>
<td>CG_72_L</td>
<td>9.5</td>
</tr>
<tr>
<td>CG_1_G</td>
<td>9.4</td>
</tr>
<tr>
<td>CG_24_G</td>
<td>9.9</td>
</tr>
<tr>
<td>CG_72_G</td>
<td>9.4</td>
</tr>
</tbody>
</table>

Table 6.4 MCV’s of each sulphate swell trial sub batches

To assess any changes to the clod grading as the mellowing periods progressed (for example caused by pulverisation from the early lime clay reactions; Figure 5.10), a 2kg random sample from each sub-batch was passed through the 5mm, 10mm, 14mm and 20mm sieves. In addition, for the CG_L batch the water content was also determined for each clay clod from these size gradations. This was undertaken to determine whether the smaller clod sizes would adsorb greater amounts of the mixing water across the numerous stages of water addition. This comparison may then allow a better understanding of how material preparation can cause heterogeneity through specimens (as discussed in Section 5.7.4).
6.2.4.2 Curing and Soaking

Following compaction, the CBR and proctor specimens were trimmed to test dimensions and the proctor samples released from the split mould. The specimens were covered with plastic wrap, sealed within close fitting containers and cured (unsoaked) at 20°C for 28 days.

The rationale for the unsoaked cure period related to thoughts on a reasonably foreseeable time delay between completion of an on-site ESLS operation and the immersion of this layer in water. As ESLS operations require an area free from standing water, and specifications i.e. (The Highways Agency 2009) require that measures are taken to protect a compacted layer from direct surface water, it is likely that a significant time delay will elapse before a treated layer becomes immersed. However, post construction changes to the groundwater regime or defects in surface water drainage may well cause this flooded scenario to develop within several months of compaction. The unsoaked curing period used in this study (28 days at 20°C) was based around an assumption that a near surface pavement layer would be exposed to temperatures around 10°C and that under reasonably foreseeable circumstances, a fully immersed conditions would not occur until 68 days after compaction. For consistency with usual practice, it was planned to use 20°C for the unsoaked laboratory curing temperature, which required the 68 days @ 10°C to be converted into an equivalent time period using a 20°C temperature. This was undertaken using the degree hours approach described in series 800 of the MCDHW (Highways Agency 2009). Degree hours are the sum of the ambient temperature >3°C for each hour of cure, which for the assumed site condition would be:

\[ 68 \text{days} \times 24 \text{hours} \times (10^\circ \text{C} - 3^\circ \text{C}) = 11,424 \circ \text{C hours} \]

Therefore to determine the number of days at 20°C:

\[ \frac{11,424 \circ \text{C hours}}{24 \text{ hours} \times (20^\circ \text{C} - 3^\circ \text{C})} = 28 \text{ days} \]
Upon completion of unsoaked curing, the specimens were weighed and measured to confirm that negligible volume change and mass loss (<0.1%) had occurred. The proctor samples were then arranged within the NST apparatus and placed to soak (appendix E). The control NST specimens were immersed in a tank of 20°C water shared with the soaked CBR’s. The NST specimens to undergo temperature reduction were placed in their own bucket of water held at 20°C (as maintained by a constant room temperature of 20°C) and after 35 days the bucket was transferred to an environmental chamber maintained at 8°C.

The CBR’s were soaked in general accordance with BSI (1990b) and the soaking phase was continued until the same criteria for the NST trial was met (i.e. <0.02mm plate swell over 3 days). As noted in section 1.6.1.2, the fail criteria for the soaked CBR is related to the vertical displacement of the swell plate. If this linear swell were perfectly uniform then the failure values of 5mm/10mm would correlate with 3.9/7.8% volume change. However, a real specimens may not swell in this perfect manner. Therefore, the volume change of the CBR’s were also determined using the volume on immersion test (BSI 1990a). Whereby, the volume of the post swell CBR specimen volume (including the mould but excluding base/swell plates) was first determined. The material which had expanded above the top of the mould was then trimmed flush and the volume of the trimmed specimen measured by immersion. The percentage difference in volume was then determined. Finally, the CBR value was then obtained for the specimen.

6.2.4.3 Post Cure Tests

Following completion of the soaking phase and to better understand how the soaking water was able to permeate through each of the CBR and NST specimens, the specimens were divided into subsamples from the regions shown in Figure 6.3. The oven dry water content (@75-80°C) of each sub sample was then determined so the water content profile across the
different specimens could be compared. Sub-samples from the NST’s were also oven dried at 40°C for mineralogy testing by XRD and thermal analysis by TGA (Section 6.4).

![Diagram showing how the proctor (left) and CBR (right) specimens were divided into sub-samples](image)

**Figure 6.3 Illustration of how the proctor (left) and CBR (right) specimens were divided into sub-samples**

### 6.2.5 Observations During Preparation

The initial mixing and addition of lime to the clay clods caused a significant change to the clod size gradations (Figure 6.4 and Figure 6.5).

For the Ay clay, compared with the 100% of the untreated clods passing the 20mm sieve, for all plots of the lime treated / mellowed material there was a significant proportion of clods (29-35%) retained on the 20mm sieve (Figure 6.4). There was also a corresponding reduction to the smaller clay clod sizes in the mellowed samples. This suggests that when the clods were initially spread out on the mixing table, some of the clods were agglomerated before the lime binder was added / mixed. The untreated clods had a water content that was 7-9% higher than the WP of 25% (Table 6.2 and Table 6.3), so this plastic behaviour is unsurprising. Once the lime binder was added, there was relatively little further change to the clod size distribution over the different mellowing periods, suggesting limited further agglomeration and
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Figure 6.4 Clay clod gradations for the different mellowing periods (Ay lime only batch)

Figure 6.5 Clay clod gradations for the different mellowing periods (CG lime only batch)
minimal pulverisation from the lime clay reactions. The former is considered additional evidence of the clod surface boundary caused by an increase to plastic limit with the early lime clay reactions (section 5.7.3). The lack of significant pulverisation with mellowing suggests that the less aggressive reaction of hydrated lime (absence of slaking heat from quicklime hydration) and regular addition of mixing water, prevented the clods from becoming friable and breaking down with mixing (Figure 6.4). The untreated CG clay was too wet / plastic to sieve, so it is not possible to observe any immediate changes between blending and lime addition. Considering the wet/plastic nature it is likely that some of the clods will have agglomerated together, however the addition of lime did then make it possible to sieve the clods by 1 hours mellowing. As with the Ay clay, the plots of the different mellowed batches are relatively closely spaced (Figure 6.5). Although, with 72 hours mellowing there is a noticeable increase in the proportion of larger clods and corresponding reduction in the proportion of smaller clods. Furthermore, the finest clod gradation (<5mm) disappears entirely from the 72 hours mellowed plot (Figure 6.5). This may be explained by the higher surface area of the smaller clods adsorbing proportionally more water (as discussed relating to specimen soaking in section 5.7.5). Figure 6.6 shows how increases to the water content with progressive mellowing stages / mixing water addition were disproportionately concentrated into the smaller clod sizes. As an example, by 72 hours mellowing the water content of the 5-10mm clods was 1.5% greater than the >20mm clods. It is suggested that at this stage, the water content of the very fine clods had increased to a point in excess of their \( W_P \) (The \( W_P \) would also increase in mellowing due to the early lime clay reactions), and allowed these fine clods to join together.

It can be summarised that changes to clod sizes across all sub-batches of the same test clay were relatively minor and due to the ongoing addition of mixing water, pulverisation was minimal/absent.
6.3 SULPHATE SWELLTRIAL RESULTS

The swell values and number of days each sub-batch spent under soaked curing conditions are set out in Table 6.5 (NST) and Table 6.6 (CBR). Execution of the swell phase of the trial ran in general accordance with the planned method and it was possible for the majority of specimens to remain soaking until each specimen in a sub-batch registered <.02mm swell over 3 consecutive days. However, due to constraints some of the Ay tests were stopped part way through the swell cycle. Due to another research project requiring the space, it became necessary to relocate all NST 8°C specimens to a smaller environmental chamber with space for only 10 specimens. So the Ay_L_24 and Ay_G_24 sub batches were stopped after respective soaking periods of 97 and 89 days. Furthermore, swelling of the Ay specimens continued for a much longer period than anticipated (242 days) and due to the EngD timescale
it became necessary to stop Ay_L_1 and Ay_L_72 sub-batches before swell had fully completed. Although the swell plate trends suggest that swelling for these specimens was in the order of 90-95% complete (Figure 6.10 and Figure 6.11).

<table>
<thead>
<tr>
<th>Sub-batch</th>
<th>Days Swelling</th>
<th>Volume change (%)</th>
<th>Days Swelling</th>
<th>Volume change (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ay_L_1</td>
<td>234</td>
<td>5.11</td>
<td>234</td>
<td>8.16</td>
</tr>
<tr>
<td>Ay_L_24</td>
<td>97</td>
<td>3.72</td>
<td>97</td>
<td>5.14</td>
</tr>
<tr>
<td>Ay_L_72</td>
<td>232</td>
<td>4.35</td>
<td>242</td>
<td>7.92</td>
</tr>
<tr>
<td>Ay_G_1</td>
<td>190</td>
<td>4.85</td>
<td>219</td>
<td>5.68</td>
</tr>
<tr>
<td>Ay_G_24</td>
<td>89</td>
<td>3.22</td>
<td>89</td>
<td>3.85</td>
</tr>
<tr>
<td>Ay_G_72</td>
<td>187</td>
<td>2.52</td>
<td>201</td>
<td>4.68</td>
</tr>
<tr>
<td>CG_L_1</td>
<td>127</td>
<td>6.44</td>
<td>127</td>
<td>8.22</td>
</tr>
<tr>
<td>CG_L_24</td>
<td>132</td>
<td>5.31</td>
<td>132</td>
<td>6.10</td>
</tr>
<tr>
<td>CG_L_72</td>
<td>82</td>
<td>4.40</td>
<td>95</td>
<td>4.22</td>
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<td>CG_G_1</td>
<td>85</td>
<td>5.55</td>
<td>99</td>
<td>5.15</td>
</tr>
<tr>
<td>CG_G_24</td>
<td>78</td>
<td>5.34</td>
<td>77</td>
<td>5.17</td>
</tr>
<tr>
<td>CG_G_72</td>
<td>89</td>
<td>4.77</td>
<td>102</td>
<td>6.28</td>
</tr>
</tbody>
</table>

Table 6.5 Swell data for the NST’s. Highlighted values show tests stopped before swell cycle complete

<table>
<thead>
<tr>
<th>Sub-Batch</th>
<th>Days swelling</th>
<th>Volume Change (%)</th>
<th>Final Swell (mm)</th>
<th>CBR value (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ave.</td>
<td>Top</td>
</tr>
<tr>
<td>Ay_L_1</td>
<td>234</td>
<td>5.89</td>
<td>8.66</td>
<td>36</td>
</tr>
<tr>
<td>Ay_L_24</td>
<td>97</td>
<td>3.93</td>
<td>4.9</td>
<td>25</td>
</tr>
<tr>
<td>Ay_L_72</td>
<td>232</td>
<td>3.40</td>
<td>5.92</td>
<td>32</td>
</tr>
<tr>
<td>Ay_G_1</td>
<td>219</td>
<td>5.19</td>
<td>6.24</td>
<td>28</td>
</tr>
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<td>17</td>
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Table 6.6 Swell test data for the soaked CBR tests. Highlighted values show tests stopped early. N/T = Not Tested.
During de-moulding of some of the CG soaked CBR’s the top of the (heaved) specimen was so soft / delicate that a significant proportion of the upper specimen (e.g. 15-20mm deep) broke away/disintegrated. This prevented the measurement of volume change and the top CBR value for 3 of the 12 test specimens (affected specimens noted in Table 6.6).

6.3.1 NST CORRELATION WITH VOLUME CHANGE

Before the results are analysed in any detail, it is first useful to evaluate the assumption that swell plate readings would correlate closely to specimen volume change. Due to the potential for the Leighton Buzzard sand layer to influence the swell plate readings, the measured volume change % was used as the primary means of assessing the degree of volume change between specimens. However, the plate readings over the test duration (Figure 6.10 to

![Graph showing correlation between swell (mm) and volume change % for NST and CBR specimens. The graph includes linear regression lines for both NST and CBR, with equations: y = 1.1324x R² = 0.8706 for NST and y = 0.7003x R² = 0.8495 for CBR.](image)

Figure 6.7 Plot of volume final plate swell readings versus volume change for the NST and CBR specimens
Figure 6.13) are informative with regard to understanding when the measured swell occurred and a reliable indicator of specimen swell. However, in Figure 6.7, there are 5-6 specimens which outline the close plot of this correlation. Figure 6.8 and Figure 6.9 compare the swell plate reading against the volume change for each individual specimen and are useful for identifying which sub-batch outlines the noted correlation. For the Ay clay the swell plate readings for the lime only batches cured at 8°C (i.e. AY_L_1; AY_L_24 and AY_L_72) associate with an apparent, but consistent over-estimation of the measured volume change in these specimens (Figure 6.8). Conversely, with the CG clay, the swell plate readings associate with some under-estimation of the measured volume change for specimens CG_L_1_8°C; CG_G_1_20°C and CG_G_1_8°C (Figure 6.9).

It can be concluded that while the relationship between swell plate readings and volume change is very strong, it is not always exact.

6.3.2 CBR SWELL CORRELATION WITH VOLUME CHANGE

A similar comparison of the final swell plate reading versus volume change % was conducted on the much smaller dataset (9 values) of soaked CBR tests (Figure 6.7). A linear correlation between the final values of volume change % against the swell plate readings is evident. A $R^2$ value of 0.85 suggests that the plots of swell plate readings over time (Figure 6.14 to Figure 6.15) are a reliable indicator of specimen swell, although again there were 3-4 outliers to the trend. Furthermore, the equation for the CBR swell linear correlation is $y = 0.7003x$ and would calculate a value of 3.5% volume change for the 5mm CBR test pass fail criteria, i.e., less than the theoretical value of 3.9% (section 6.2.4.2). This difference may be substantially explained by the loss of some of the untrimmed CBR specimen before / during the volume on immersion test. The material which heaved out the top of the mould was relatively delicate/loose (especially at the edges), and some of this broke away during demoulding. This
Figure 6.8 Swell plate readings and volume change per specimen (Ay)
Figure 6.9 Swell plate readings and volume change per specimen (CG)

Figure 6.10 NST 20°C swell plots for Ay. The 24 hour mellowing tests were stopped early.

Figure 6.11 NST 8°C swell plots for Ay. The 24 hour mellowing tests were stopped early.
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Figure 6.12 NST 20°C swell plots for CG.

Figure 6.13 NST 8°C swell plots for CG.
Figure 6.14 Soaked CBR swell plots for Ay. The 24 hour mellowing tests were stopped early.

Figure 6.15 Soaked CBR swell plots for CG.
effect would cause the apparent volumetric swell to appear lower than it was and may explain the loss of the relationship. Therefore, for the CBR’s, the swell plate values are considered the more reliable indicator of volumetric swell.

The noted differences in swell plate reading and volume change highlight potential issues with the degree of repeatable accuracy and / or challenges with not having repeat specimens for each the sub-batches in both the CBR and NST methods. However, the observed differences are small enough to allow careful comparison between sub batches in the manner intended.

It is also worth noting that for any change in swell plate reading, the volume change of the NST specimens is significantly larger than the equivalent volume change in the CBR test (i.e. for the NST 5mm swell \(\approx 5.67\%\) volume change; Figure 6.7). So, the swell plate readings from the NST and CBR tests are not directly comparable.

6.3.3 **RESULTS ANALYSIS**

Prolonged soaking caused a considerable volume change i.e. up to >8% in both clay types. Although influences from curing temperature, binder type, clay type and mellowing caused substantial differences to the overall range of values reported (2.5%-8.2%) and these influences are analysed (Table 6.5 and Table 6.6).

6.3.3.1 **Temperature influence**

For all (CBR and NST) tests undertaken at 20°C, the initial rate of sample swell was relatively high and then reduced at an inversely proportional rate until swelling stopped. For all 8°C specimens, the same inversely proportional swell relationship is present overall, however, there was a noticeable change to the swell rate which occurred promptly after the reduction from 20°C to 8°C at 35 days soaking. For the majority of specimens this change was an increase,
however there were three specimens (CG_72_L; CG_G_1 and CG_G_24) where little change, or perhaps small reductions to the swell rate were identified (Figure 6.13).

With the overall influence of temperature noted, the further observations are summarised for each test clay.

### 6.3.3.2 Aylesbury

For the CBR and NST specimens cured at 20°C, there is a reduction in the degree of swell associated with the longer mellowing periods. Furthermore, the GGBS specimens show less swell than their lime only equivalents. Therefore, for the 20°C specimens, the highest swell values were recorded in the Ay_L_1 specimens (NST = 5.11%; CBR = 8.7mm) which were still swelling when the trial was stopped. The lowest swell was recorded in the Ay_G_72 sub batch (NST = 2.53%; CBR = 3.2mm).

For all sub-batches, the NST 8°C specimens record significantly larger values of swell than their 20°C equivalents and this increase was most significant for the lime only specimens. Furthermore, while mellowing lessoned swell in the 20°C lime only batches, there is only a small difference between the (still swelling) values recorded for 8°C Ay_L_1 (8.2%) and Ay_L_72 (7.9%).

For the GGBS specimens, although the temperature reduction does cause a clear change in swell rate, the increase is smaller than recorded for the lime only specimens (Figure 6.11). Furthermore, the increased swell caused by temperature reduction appears to be the same for all mellowing periods (i.e. the step in the swell plots after 35 days is the same for all GGBS plots (Figure 6.11). The final swell values show that the specimens with longer mellowing had less total swell (Ay_G_1 = 5.7%; Ay_G_72 = 4.8%). However, inspection of the swell plot (Figure 6.11) shows that this difference in swell occurred during the 35 days when soaking was
at 20°C. This suggests that for both the lime only and GGBS batches, while longer mellowing appears to reduce the degree of swelling in samples cured at 20°C, mellowing appeared to have little influence on swell processes operative at 8°C.

6.3.3.3 CaulGyp

For the lime only specimens, the use of a longer mellowing period corresponds with a reduction in the degree of swell for all tests and at all temperatures. For the 20°C tests, the degree of swell was the highest in the CG_L_1 specimens (NST = 6.5%; CBR = 9.4mm) and the lowest in the CG_L_72 specimens (NST = 4.4%; CBR = 5.5mm).

The GGBS specimens were much less sensitive to changes in the mellowing period, especially at 20°C where the range of results are closely spread (NST 4.8-5.5%, CBR = 5.6-6.6mm). At 20°C, there appears to be some marginal benefit to longer mellowing periods, as the lowest swell values from both NST and CBR tests were from the CG_G_72 sub-batch. For the NST 8°C specimens, the influence of mellowing duration is reversed with swell from CG_G_72 actually increasing to 6.2%. This increase in swell rate for CG_G_72 is clearly seen from 35 days in Figure 6.13, whereas the swell plot of Ay_G_1 and Ay_G_24 are little affected.

6.3.4 Comparison of NST with CBR results

6.3.4.1 Swell rate

At trial outset, it was hypothesised that early access to water around all sides of a specimen of smaller volume would cause the swell cycle in NST specimens to complete much sooner than the equivalent soaked CBR. While there were some variations, typically there was not a substantial difference between the swell duration in the CBR and 20°C NST specimens from each sub batch (Table 6.5 and Table 6.6). The greatest difference was for the CG_L_1 and
CG_L_24 specimens, where the opposite of the expected effect was seen and CBR specimens completed their swell cycle 33-35 days, (or 36% sooner) than the NST tests. This suggests that the test method may have some influence, but is not a primary control over the swell cycle duration at 20°C.

For the Ay specimens, while the swell durations may be similar (Table 6.5 and Table 6.6), the initial rate of swell in NST specimens was at a significantly greater rate than for CBR specimens (Figure 6.10 and Figure 6.14). This difference was less clear for the CG specimens. As an example, with the NST for sub-batch Ay_G_72, 50% of the final swell value was realised within 32 days, whereas a much slower early swell rate in the CBR test required 57 days to achieve 50% of its final swell value. This suggests that where the swell rate is slow due to the slow supply of an ettringite reactant (i.e. water) the NST may be of potential use as an advance indicator of the degree of final swell (see further research suggestions in section 7.4)

6.3.4.2 Water content profiles

Comparison of the water content results from the different locations across test specimens show some differences between the CBR and NST tests (Table 6.7 and Table 6.8). Figure 6.16 is useful in visualising representative results from Ay_L_72 and Ay_G_72 specimens. The most noticeable aspect of the results is that the top 20mm of all test specimens were of substantially higher water content than the rest of the specimen (Figure 6.16). This is likely due to the lack of confinement at the specimen surface and the formation of weaknesses (micro-cracks) within the expanding mass exaggerating water ingress at this location. As a real pavement environment would have an overlying layer, which would cause some confinement, the extent of softening in these test specimens may be unrealistic. Thus, the very low CBR values associated with the sample top may be misleading (see Section 6.3.5).
### Table 6.7 Post cure water contents (Ay)

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### Table 6.8 Post cure water contents (CG)

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Table 6.7 Post cure water contents (Ay)

Table 6.8 Post cure water contents (CG)
Figure 6.16 Bar plot of water contents across CBR and NST specimens. Select examples shown

In comparison with the specimen top, water profiles through the rest of the specimen were much lower and relatively consistent, suggesting that the specimen had reached a steady state throughout. However, some differences are apparent. The water content throughout the CBRs are noticeably lower than the comparable NST 20°C values, especially in the Ay specimens where the average water content of the CBR’s is typically 2.6% lower. This difference is most noticeable at the sides of the Ay specimens where the water contents from the NST 20°C sub batches are an average 3.5% higher than the CBR’s. Therefore the CBR mould does appear to limit water ingress into the specimen as suggested by Notman (2011). Where a reduction in temperature was associated with an increased volume change, as expected, the NST 8°C water contents align with an increase in value from the 20°C equivalent (Table 6.7 and Table 6.8).
6.3.5 CBR VALUES AND HA 74 PASS/FAIL CRITERIA

Where it was possible to obtain a CBR value from the top and bottom of a specimen, the average CBR value from all sub batches was above the 15% required for a capping specification in accordance with HA74 (The Highways Agency 2007). In all cases, the CBR values from the top of specimens are substantially lower than values obtained from the base. The much greater degree of water ingress at the top of specimens (see Section 6.3.4.2) is the likely cause of this reduced CBR value and reason that three of the CG specimens broke up during demoulding and could not be tested. As the CBR values are relatively high (typically higher than those achieved in the nucleation trial; Section 5.6.2), this indicates that while the lime – High TPS clay soil reaction caused significant swell, it has also caused a significant increase in strength, stiffness or deformation properties.

While the lack of repeat tests limits the value of any observations relating to differences in CBR value between the sub-batches, it appears that the specimens from the shortest mellowing periods achieved higher values than those from the longer mellowing periods. Furthermore, and contrary to expectations, CBR values from the GGBS specimens are not greater than their lime only counterparts, and in several incidences the GGBS CBR values are actually lower than lime only equivalents.

If the non-standard unsoaked cure period (28 days) is ignored, an average soaked CBR value of 32% and 3.2mm swell for sub-batch Ay_G_72 would have passed the HA74 specification. The NST 20°C test for Ay_G_72 recorded a very similar degree of volume change (2.5%) to concur with the CBR swell values. However, with volume change of 4.7%, the NST 8°C underwent 85% more expansion than the 20°C equivalent and if viewed in terms of equivalent CBR swell value would have failed the HA74 specification. This discrepancy highlights the importance of undertaking swell tests at site realistic temperatures and
understanding the different swell process operative at 8°C. The latter is considered further with mineralogical testing (see Section 6.4).

### 6.4 XRD AND TGA

The data presented in this section are from early results. The research started by the EngD study is continuing beyond the thesis submission. The XRD and TGA undertaken up to point of submission identified mineralogical relationships which may assist explanation of the swell processes observed. However, an expansion of this work is planned to include repeat tests; tests on specimens not yet tested and wider temperature scan ranges for the TGA. Therefore, this section presents suggested reasons for some of the mineralogical processes behind the observed sulphate swell behaviour, to be published later.

#### 6.4.1 ANALYSIS UNDERTAKEN

Table 6.9 confirms which sub-samples were analysed by each technique and for easy comparison the associated volume change is also shown. The combinations analysed cover the greatest and least volume change recorded in both clay types, along with the range of mellowing periods and binders used. To determine changes caused by the binders over time, sub samples from the natural clay and from select mellowing periods (MELL) were also analysed.

#### 6.4.2 METHOD

The analyses were undertaken in the Chemistry department of Nottingham Trent University. Before analysis, samples were dried at 40°C and ground using a pestle and mortar to pass a 212μm sieve.

For XRD, samples were examined using a Philips X’Pert Pro powder diffractometer in Bragg Brantano geometry. Samples were mounted as powders on disks that were spun at 4
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Table 6.9 Summary of sub-samples analysed by XRD and TGA. Red values indicate specimen was still swelling when test stopped (as explained in Section 6.3)

Rotations per minute to reduce preferred orientation effects. Diffraction patterns were recorded using Ge(111)-monochromated Cu_kα1 radiation over a range of 5-50 degrees of 2θ. For TGA, samples were analysed on a PerkinElmer 4000 Thermogravimetric analyser. 20-50mg powder samples were investigated over 35°C – 500°C at a heating rate of 10°C/min, in an atmosphere of nitrogen flowing at 30 ml / min.

TGA is useful as a means of phase identification as different minerals will typically undergo mass loss over characteristic temperature ranges. The results in this study are presented as derivative curves (DTA) which assists with identifying temperatures where changes in the rate of mass loss were more/less substantial. Table 6.10 summarises the literature used to align phases with mass loss over specific temperature ranges in this study. As the derivative curves were normalised to an initial sample mass of 50mg, comparison between
curves allows an appraisal of which specimens contain more/less of the mineral associated with the temperature range. Thus, DTA has further value as a semi-quantitative technique.

It was anticipated that XRD would assist with confirming the presence of either ettringite or thaumasite. However, as the characteristic basal spacing’s of ettringite and thaumasite are very close, it is not usually possible to differentiate between these phases using XRD alone (Thaumasite Expert Group 1999). TGA is more appropriate in this regard as the hydrated water molecules are released over different temperature ranges for each mineral. Ettringite is associated with a rapid loss from 50°C, whereas with thaumasite the rapid loss starts from 110°C (Taylor, 1990). One challenge with using TGA to assist with identification of cementitious compounds is that the temperature range of mass loss from C-S-H may overlap that of ettringite (Table 6.10). Therefore, when both phases are present this may cause some subjectivity as each may influence the DTA peak height. However, as the range of temperature corresponding with mass loss do not match each other exactly, changes to the peak profile should indicate a relative change in the amount of these phases between samples.

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<td>Rapid loss from 50°C (TGA) Mass loss centred at: • Early cure 65°C (DTA) • Late cure 90-100°C (DTA)</td>
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<td>Thaumasite</td>
<td>Rapid loss from 110°C (TGA)</td>
<td>(Taylor 1990)</td>
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Table 6.10 Summary of literature used for characteristic temperature ranges for mass loss during TGA/DTA
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Figure 6.17 XRD traces for Ay sub-samples. Key peaks are shown (Ett = ettringite; Gyp = gypsum)

Figure 6.18 XRD traces for CG Sub-Samples. Key peaks are shown (Ett = ettringite; Gyp = gypsum)
Figure 6.19 DTA curves CG_L sub-samples. The occasional spikes in the CG_Untreated are from electromagnetic interference during data capture.

Figure 6.20 DTA curves for CG_G sub samples
Figure 6.21 DTA curves for Ay specimens with greatest and least volume change. The occasional spikes in the Ay_Untreated are from electromagnetic interference during data capture.

Figure 6.22 DTA curves comparing 20°C and 8°C plots of Ay specimens. To highlight temperature influence, the 20°C (red) and 8°C (blue) for each sub-batch are shown overlapping.
6.4.3 **RESULTS ANALYSIS - CAULGYP**

Comparison of XRD traces for the untreated clay and all lime and GGBS sub batches reveal two consistent changes (Figure 6.18). The untreated clay shows strong reflections associated with gypsum, which are then absent in all cured materials. All of the cured batches have new reflections which associate with either ettringite or thaumasite (Figure 6.18).

The DTA curve for the untreated clay shows a peak approximately centred around 120°C which is consistent with the gypsum known to occur in the specimen (Figure 6.19; Table 6.10). This peak is not apparent in cured specimens which instead feature large peaks associated with a mass loss starting from around 55°C and approximately centred between 87-100°C (Figure 6.19 and Figure 6.20). Otherwise, there is little difference between the untreated and cured specimens. The disappearance of gypsum and mass loss from 55°C is consistent with ettringite growth (Taylor 1990). The mellowed specimens do feature the gypsum peaks approximately centred at 120°C, although they are of reduced intensity from the untreated clay (Figure 6.19 and Figure 6.20). The mellowed samples also have a pronounced mass loss starting from 55°C, however, the peaks heights are much lower than the cured sample and are centred on a lower temperature (73-83°C) which is consistent with early cure ettringite (Table 6.10). The 1 and 24 hour mellowed specimens feature a wide and low peak between 430°C-455°C which is likely from the added lime (Table 6.10). However, this lime peak is very weak/absent in both 72 hour mellowed specimens and absent for all cured specimens (Figure 6.19 and Figure 6.20).

The XRD and DTA data are consistent in demonstrating that lime addition to the CG clay reacts with gypsum to form ettringite, a process which commenced during mellowing. The quantity of ettringite formed in the MELL72 sample is much less than quantities present in the fully cured batches. When comparing the DTA curves for the different sub batches of cured
specimen, there is little difference in the intensity of the ettringite peaks. This, suggests that by completion of specimen swell, a very similar quantity of ettringite is present across all the specimens and that ettringite growth in the CG continued until the supply of sulphate (from the gypsum) was exhausted. Notably, while the 8°C temperature did change the degree of volume change, the quantity of ettringite formed did not change.

The similarity between Lime only and GGBS DTA curves (Figure 6.19 and Figure 6.20), suggests GGBS did not cause a substantial quantity of another mineral to form, or reduce ettringite growth. However, as C-S-H is a primary product of the lime/GGBS reaction (Wilkinson et al., 2010) it is difficult to be certain that some of the large peak attributed to ettringite is not from C-S-H (Table 6.10). Although as the peak profiles of the GGBS and lime specimens are closely comparable (i.e., no broadening/narrowing or step changes), it does not seem likely that GGBS caused a substantial change to the mineralogical composition as analysed.

6.4.4 RESULTS ANALYSIS – AYLESBURY

The Ay results (Figure 6.17, Figure 6.21 and Figure 6.22) are generally similar to the CG, in that lime treatment and curing causes gypsum from the natural clay to diminish and ettringite to grow, although there are some significant differences between the sub-batches.

For the XRD data, gypsum reflections are strongest in the Ay_L_Mell72, but are also present with moderate intensity in all cured sub-batches except for Ay_L_1_8°C where reflections are very weak (Figure 6.17). The DTA curves also indicate gypsum was present in all cured specimens (Figure 6.21 and Figure 6.22)

Comparison of DTA curves of samples at the same temperature suggests there is an overall relationship between increased quantities of ettringite and an increased degree of swell measured in the Ay samples (Figure 6.21; Table 6.9). However, as the mellowing period and
temperature change also influence the degree of swell (Figure 6.22), it is not a straightforward comparison. Compared with the lime only sub-batches, the peaks associated with ettringite are lower in all GGBS sub-batches, which generally experienced less swell. This is most pronounced when comparing the large ettringite peak of Ay_L_1_20°C (5.1% volume change) with the much smaller ettringite peak in Ay_G_72_20°C (2.5% volume change; Figure 6.21; Table 6.9).

With regard to the influence of temperature reduction, while the measured volume change was consistently greater at the lower temperature for all sub-batches, the ettringite peaks of the 8°C specimens are noticeably smaller than the 20°C equivalents (Figure 6.22). This is with the possible exception of the Ay_G_1 specimens, where ettringite peaks in the 20°C (4.9% volume change) and 8°C (5.7% volume change) specimens are closely comparable. Therefore, the extra volume change associated with the lower temperature, does not appear to have been caused by the formation of further quantities of ettringite. When comparing samples of the same temperature, there does appear to be a relationship between mellowing and the quantity of ettringite formed. Specimens mellowed 1 hour have larger ettringite peaks than the 72 hour equivalents (Figure 6.21).

6.4.5 SUMMARY

The XRD and semi-quantitative TGA data indicate that volume change in both specimens is associated with ettringite growth and an associated decrease in the quantity of gypsum. For all cured CG specimens the gypsum appears to have been fully consumed upon cessation of specimen swell, whereas, the Ay specimens typically still have gypsum available.

For Ay specimens, the quantity of ettringite identified is related to the degree of sample swell and when considering samples soaked at the same temperature, greater amounts of ettringite associate with a higher degree of volume change. Furthermore, both the partial
substitution of GGBS and longer mellowing periods appear to have reduced the quantity of ettringite and associated volume change for Ay sub-batches. Whereas, with the CG, the quantity of ettringite is similar in all specimens and quantities formed are not clearly linked to volume change. Notwithstanding, GGBS use and longer mellowing did reduce the degree of swell in the lime only CG batches. All the further swell associated with the temperature reduction is not from additional ettringite growth and appears to result from a different process. The 8°C CG sub-batches treated with GGBS illustrate an increase in swell reduction with further mellowing and the similarity in the mineralogy of all CG specimens does not assist with an explanation for this anomalous behaviour.

6.5 SULPHATE SWELL TRIAL DISCUSSION

To lead the discussion, the sulphate swell trial results are considered against the strategies for sulphate swell reduction considered in section 6.1. Prior to this, a comment is made relating to the apparent relatively homogenous distribution of ettringite throughout the clay clods.

6.5.1 ETTRINGITE DISTRIBUTION THROUGH CLAY CLODS

At the outset of the experimental work, one of the key questions related to whether the use of large clay clods would lead to a heterogeneous the distribution of reaction products (section 4.2.1). The nucleation trial identified that, with curing of lime treated low TPS clays, the diffuse growth of sub-microscopic C-S-H throughout clay clods led to an apparently homogenous increase to strength within individual clods (see Section 5.7.2). A high water content and saturated inter-aggregate porosity was considered key to this efficient diffusion.

While the distribution of ettringite over the individual clods was not directly investigated in the sulphate swell trial, the sub samples for XRD and TGA were obtained randomly and could have been from any location across the large clods. Therefore, it is striking
that observations of different ettringite quantities across Ay sub-batches (Section 6.4.4) and the uniformity of ettringite quantity across the CG sub-batches (Section 6.4.3) are apparently insensitive to their location across the clay clod. While the planned repeat TGA tests on select specimens would be useful to confirm this observation, it does appear that ettringite distribution across clay clods is relatively uniform. Therefore, the high water contents targeted in preparation appear to have been successful in the widespread diffusion of ettringite reactants across the clods. The widespread nucleation of ettringite is considered favourable in minimising swell as it promotes wider distribution of smaller ettringite crystals, thus avoiding expansion ‘hotspots’ (Little et al, 2010).

6.5.2 LIMITING REACTANTS

As a principal observation for this trial, it is worth noting that the time required for all specimens to complete their swell cycle was substantial. This suggests that ettringite nucleation / growth under soaking conditions, was generally very slow and the slow supply (e.g. dissolution and or diffusion) of one of the reactants may explain this slow rate. Furthermore, the Ay specimens took much longer to complete their swell cycle than the CG, and some of the lime only specimens were still swelling when the trial was stopped after more than 232 days soaking (Table 6.5). This indicates that the supply of the limiting reactant was even slower in the Ay clay. A possible explanation for this is the different properties of the natural Ay clay compared with those of the artificially prepared CG clay. The gypsum added to the reconstituted CG clay was in the form of a fine powder of a high surface area and evenly blended throughout the clods. In comparison, the natural gypsum in the Ay clay occurred as large (e.g. up to 15mm) crystals, of heterogeneous size, shape and distribution, and of substantially lower surface area than the powdered gypsum. The slower rate of gypsum dissolution and release of sulphate into the pore water in the Ay sub-batches may well be a primary reason for the difference in swell rate
between the two clay types. Furthermore, as both clay types were formed with a similar distribution of large clay clods (Figure 6.4 and Figure 6.5), the generally slow rate may also be a function of the slow diffusion of water deep into these clods. As the CG clods had been reconstituted from <500µm fragments of the source clay this will likely have introduced a structure around these fragments, perhaps causing the CG clods to be of significantly higher permeability than the Ay clods. Therefore, the migration of Calcium, hydroxyl groups, sulphate and water through the CG clods would have been less tortuous, thus facilitating an accelerated rate of ettringite growth. However, as neither an assessment of the clay pore size distribution or clod permeability was attempted, it is not possible to substantiate this suggestion of greater permeability through the CG clods.

The absence of gypsum and very similar quantities of ettringite present in all cured CG specimens indicates that ettringite growth and swelling continued in these specimens until all the available sulphate was consumed. Therefore, it is likely that sulphate was the limiting reactant in CG sub-batches. As significant gypsum was still present in the post cure Ay specimens, sulphate does not appear to have been the limiting reactant. As the partial substitution of GGBS for lime caused a notable reduction in the quantity of ettringite formed in the Ay specimens, it is likely that calcium became the limiting reactant for ettringite growth in these samples. This appears to partially explain the lesser degree of swell in GGBS specimens, however, there are further factors to consider in this regard (see Section 6.5.4).

6.5.3 CONTROLLED ETTRINGITE GROWTH

For most sub-batches from both clay types, the use of extended mellowing periods does generally associate with a reduction in specimen swell, suggesting that this strategy typically did work as theorised (Section 6.1.2). The noted exceptions were the NST 8°C cures from the Ay_L and CG_G sub-batches, where the influence from temperature reduction appeared to
impart a more significant change to swell than the mellowing period alone (as separately discussed in section 6.5.5).

The TGA analysis for both clay types does indicate that some ettringite growth did occur during mellowing (see sections 6.4.3 and 6.4.4). However, even with 72 hours mellowed samples from the more reactive CG clay the majority of gypsum was undissolved at the point of specimen compaction. Comparison of DTA curves from MELL1 and MELL72 samples demonstrates there was not a substantial difference in the quantities of ettringite / gypsum present (Figure 6.20). This is further evidence that the slow dissolution of gypsum is a rate defining factor in ettringite formation in clay soils (section 6.5.2). It also suggests that, due to a similar quantity of sulphate supply from the slowly dissolving gypsum, upon compaction the degree of ettringite nucleation / growth will have been similar for all mellowing periods. Therefore, it is suggested that the addition of extra mixing water during the prolonged mellowing periods is the primary reason for reduced volume change in these sub-batches. The extra mixing water enabled a higher proportion of non-expansive ettringite growth from water internal to the clay soil system (Little et al. 2010). It is also likely that the extra water content in the 72 hour mellowing specimens would have enabled a higher degree of gypsum dissolution and non-expansive ettringite growth during the 28 days of unsoaked curing. However, as mineralogical analysis was not attempted at an interim point between unsoaked and soaked curing, this cannot be substantiated.

6.5.4 CHEMICAL BALANCE

As noted in section 6.5.2, partial substitution of GGBS for lime did typically associate with a reduction to specimen swell. However, as comparison of TGA and XRD analysis undertaken on lime only and GGBS sub-batches did not identify any different phases (either before sample compaction or after soaked cure), it has been suggested that the lower calcium content was the
primary reason for the reduced expansion. However, it is possible that an alternative mineralogy did form, but the quantities were too small to be revealed by XRD or TGA techniques. Furthermore, if the addition of GGBS did cause the rapid and localised formation of C-S-H, and/or another mineralogy at the periphery of clay clods, this may well have caused a reduction to permeability at this location (as suggested in section 6.1.4). This would also provide a satisfactory explanation for the lower water content and ettringite growth in the Ay_G specimens. For the CG_G specimens, the same explanation could still apply, however, the lime/GGBS reaction products may have been ineffective in ‘blocking’ a larger pore space between reconstituted clay fragments (structure as suggested in section 6.5.2). A structure of increased permeability caused by artificial preparation may substantially explain why the CG specimens did not clearly benefit from the use of GGBS, whereas the natural clay did. Therefore, in this regard the mineralogical results are subjective and it remains a possibility that GGBS use did cause localised changes to the mineralogy that significantly influenced specimen swell.

6.5.5 TEMPERATURE INFLUENCE

It is clear that the reduction in temperature caused substantial changes to the degree of volume change and this typically caused extra swell in most specimens, particularly the lime only sub-batches. As the mineralogical data suggests that temperature reduction did not cause thaumasite growth, nor the formation of further ettringite, a separate explanation for the increased expansion is required. Observations from Wild et al.’s (1993) study may assist with this alternative hypothesis.

Wild et al (1993) subjected lime treated kaolinite spiked with gypsum to curing regimes of different unsoaked / soaked durations. They identified that specimens with the highest degree of sample swell did not correlate with the greatest quantities of ettringite formed and
instead the highest degree of swell actually associated with less crystalline ettringite. From these findings Wild et al (1993) proposed that the very high degree of swell occurred prior to the precipitation of crystalline ettringite and was associated with an ettringite precursor. The ettringite precursor was described as a colloidal gel with a high affinity to imbibe water molecules, sourced from the soaking water. It was suggested that osmotic swelling of the colloidal gel would prevent crystalline ettringite growth, and this condition would remain as long as a continuous supply of water was available. Without a continuous supply of water, the swell curtails, allowing the reaction to complete and form crystalline ettringite, at which point the system becomes volumetrically stable (Wild et al, 1993).

For the present study, the ettringite precursor may assist explanation of the increased swell at lower temperatures. If it is accepted that swell is associated with the ettringite precursor imbibing water (Wild et al, 1993), it is logical to suggest that when conditions allow crystalline ettringite to precipitate from the precursor then swell will stop. In this regard, while the enthalpy of reaction would associate lower temperatures with a higher degree of ettringite precipitation (Perkins and Palmer, 1999), the lower temperatures would slow the diffusion of reactants. This may be a particular issue with the heterogeneous nature of a lime-clay soil system, where the reactants must diffuse along a tortuous path via pore channels within a soil structure (see Section 5.7.2). Thus, at a lower temperature the slow rate of diffusion would slow the rate of ettringite precipitation from the precursor. This would cause the ettringite precursor, with high affinity to draw water in from the soaking water to remain in this state for longer. This explanation would meet with observations that the 8°C specimens; swelled for longer; were of higher (oven dry) water content; underwent greater volume change, yet associated with the same or a lesser quantity of ettringite than their 20°C equivalents (Figure 6.19 to Figure 6.22).
The results from the 8°C CG_G sub batch are anomalous to the behaviour expected from theory and the rest of the results achieved in this trial. It is difficult to apply a satisfactory explanation as to why the longer mellowed specimens underwent further swell with temperature reduction, and the sub batches of shorter mellowing duration actually underwent less swell than 20°C equivalents. It is suggested that further research into this would be required to better understand the influence of longer mellowing periods on lime with GGBS treatments at lower temperatures.

6.5.6 INDUSTRY RELEVANCE

6.5.6.1 Identifying swell risk with laboratory tests

As a key finding, this trial has identified that swell mechanisms operative at site relevant temperatures (e.g. 8°C) are different to those at 20°C. Where soaking water is available, the lower temperature causes the lime-clay soil system to remain in a condition where water is drawn into the system for a substantially longer period. This study concurs with other studies (Wild et al., 1993; Little et al., 2010) that prolonged unsoaked curing before immersion allows time for non-expansive ettringite to form from water available within the soil pore water and that this reduces subsequent expansion during soaking. This highlights the importance of a site relevant curing regime in any standard test and key questions relate to a suitable period of unsoaked cure before water immersion and the temperatures used for both unsoaked and soaked curing.

The experimental work for this study included an unsoaked cure period of 28 days at 20°C. This was based upon the assumption that a degree hours conversion (The Highways Agency, 2009) of 68 days at a site relevant temperature of 10°C would match with a reasonably foreseeable delay before the immersion of a compacted SLS layer (see section 6.2.4.2). There
are two potentially significant issues with this assumed method and these are discussed. First, it could be argued that certain site conditions may cause the treated layer to become immersed sooner than the 68 days chosen and for the purpose of a standard test, it may be most relevant to consider possibility rather than probability. Notwithstanding, an extended unsoaked cure period appears to be of such key significance, the very short periods in the existing standard tests (i.e. 3 days @20°C) may appear unreasonably short? A second issue relates to the use of the degree hours conversion to determine the equivalent cure period at 20°C. The degree hours conversion assumes that increases in time and temperature are a linear function, however, this study has identified that the influence of temperature on ettringite precipitation in a heterogeneous clay soil system is complex. The reduced energy and rate of diffusion in a low temperature cure environment may constrain ettringite growth in a non-linear manner. It has been suggested that the more substantial degree of volume change is associated with the ettringite precursor, and that the precursor would remain stable for a much longer time at lower temperatures. Therefore, 28 days unsoaked curing at 20°C would have the potential to cause a much greater degree of non-expansive ettringite growth than 68 days unsoaked curing at 10°C.

This observation of temperature effects may be extended to a potential explanation as to why laboratory swell tests (at 20°C) undertaken for reported failures (e.g. the M40) determine much smaller degrees of volume change than were realised on site. The use of higher temperatures, (especially the 40°C soaked cure temperature of the accelerated swell test; (BSI 2004b) may substantially reduce the time that the ettringite precursor remains stable, and the site relevance of this environment may be extremely low. So, without further research into this (see recommended research in section 7.4), the validity of using a temperature higher than 10°C at any point during a swell test must be strongly questioned.
Therefore, this study recommends that a site relevant swell test requires cure temperatures which meet with those anticipated on site and future adaptations to tests should consider using unsoaked and soaked cures temperatures of <10°C.

6.5.6.2 Hydrated Lime

The results show that the sulphate swell rate is fundamentally restricted by the slow supply of soaking water. Large clay clods can be considered as a primary defence against sulphate swell, they provide a tortuous pathway for soaking water and even in a laboratory swell test where soaking water was provided from all sides the rate of swell was very slow. The use of hydrated lime in lieu of quicklime also appeared to promote several benefits in this regard. The absence of heat (from quicklime slaking) is suggested to have slowed the initial lime clay reactions; limiting pulverisation and preventing a rapid gain in clod strength during mellowing. The latter, combined with substantial mixing water, ensured clod ductility (MCV = 10) during compaction minimising inter-clod porosity and permeable pathways through the specimen. Thus, with regard to reducing risk against sulphate swelling the combination of hydrated lime/GGBS, on a fill compacted at an MCV<10 is recommended.

6.5.7 Suitability of NST

The NST introduced in this study has a potential role as a site relevant test. The need to include a lower temperature in any site relevant test has already been recommended. The access of water to all sides appears to be of particular relevance where the overall specimen permeability is very low. As low ‘inter-clod’ permeability is recommended as a primary defence against sulphate swell (section 6.5.6) then a swell test with the capacity to test such a specimen in a timely manner is important. The mould used for the soaked CBR test does appear to limit the
rate of water access into the specimen, however, the main disadvantage appears to be that the early swell rate is much slower than the NST equivalent. In this regard the NST has greater potential use as a predictive test i.e. the final swell may be estimated from the swell plate response over the first 28 days, or possibly sooner (14 days?). However, as the use of Leighton buzzard sand material may influence the reliability of correlating the swell plate readings with volume change, some adaptations are recommended e.g. a purpose made mould / test apparatus that provides lateral confinement and water access. Further research to refine the NST or adapt the soaked CBR test is recommended (see Section 7.4).

The study results also indicate that where swelling specimens are unconfined, the expansion will impose weakness in the clay soil and a greater quantity of water will be absorbed (Section 6.3.5). A near surface environment will have a low confining stress to all sides and an unconfined swell test may be unrealistic. In addition to the issues with the high test temperatures (6.5.6), this is cause for caution when considering the site relevance of the accelerated swell test (BSI, 2004b). However, this study has not directly evaluated this test method.
7 FINDINGS & IMPLICATIONS

7.1 THE KEY FINDINGS OF THE RESEARCH

A focus on laboratory experimentation using a site relevant preparation and test parameters has identified several processes which substantially influence the engineering properties and durability to soaking water of high and low TPS soils treated with SLS.

A fundamental discovery relates to the application of lime binder to the surface of clay clods. This instigates a very rapid increase in the plastic limit at the surface of clods and neighbouring clods are not able to join together during subsequent compaction (see Section 5.7.3). Thus, field applications of lime stabilised clay results in a material discontinuum, where

- The physico-chemical behaviour of each individual clay clod is independent to its neighbours (Section 5.7.2)
- Each clay clod is separated from its neighbours by an inter-clod porosity (Section 5.7.4).

This discontinuous nature will lead to the heterogeneous development of strength throughout a compacted lime stabilised clay. The strength of each individual clay clod will increase as surface added lime dissociates into $\text{Ca}^{2+}$ and hydroxyl group and these ions migrate deep into the clod to react with the clay minerals to cause diffuse cementation (See Section 5.7.2).

The inter-clod porosity will remain as a long term weakness and efforts to minimise this porosity during compaction are particularly important for the long term durability of SLS material. The study has discussed how the use of quicklime with longer mellowing periods can cause the clay clods to develop high strength before compaction (Section 3.4 of Paper 1). The high strength clods then resist compaction and the degree of inter-clod porosity in the compacted mass will increase. This becomes a particular issue if the treated material becomes
affected by surface or ground water ingress (see Section 5.7.5). A high degree of inter-clod porosity acts as a high permeability conduit, increasing permeability through the mass. With low TPS soils the soaking water softens the treated material. With a high TPS soil the ingress of significant water way cause substantial sulphate swelling to develop.

It is suggested that for specific applications, (e.g. medium – high plasticity clay soils and especially where there is risk of a high TPS value), it may be pertinent to condition the material in a manner which optimises diffuse cementation and minimises inter-clod porosity. This requires the use of hydrated lime, which is removed from the current UK approach to lime stabilisation which almost exclusively uses quicklime. The advantage of this approach would be less inter-clod porosity and greater resilience to water ingress. A disadvantage to this suggestion is that the resultant larger clods would be of a lower immediate strength. While diffuse cementation through the larger clods would be optimised and the lower strength recovered with curing, this may not be suitable to all applications.

In relation to the use of adapting working methods to reduce the risk of sulphate swell, this EngD finds agreement with other studies that prolonged mellowing periods, with regular addition of mixing water reduces the degree of expansion from post compaction soaking. The mellowing period allows time for the lime modification reactions to progress, with the increasing plastic limit allowing further mixing water to be added. With a greater amount of water added before compaction, ettringite is able to form from water internal to the soil system and there is less expansion through the ingress of external water. The use of GGBS (as a partial substitution for the lime binder) significantly reduced the degree of sulphate swell in high TPS clay soils of natural origin, however, the influence on artificially prepared clay soil was ambiguous. The artificial preparation may have increased the permeability of this clay soil and the relevance to the site scenario is not clear.
A most significant finding relates to trial results demonstrating that sulphate swelling occurs at a faster rate and to a greater extent when a site relevant temperature i.e. 8°C, is used for soaked curing, instead of the 20°C (or higher) temperatures specified in standard swell tests (i.e. BSI 2004b, BSI 2004a). The increased swell rate has been linked to an ettringite precursor with a high affinity to imbibe water into the soil from the soaking water. It is hypothesised that the slower diffusion of ettringite reactants at 8°C would slow the precipitation of crystalline ettringite. This would cause the ettringite precursor to remain stable, and in the condition which imbibes a high degree of water into the soil, for a longer time period. Undertaking laboratory swell tests at a substantially higher temperature than the field condition may avoid the full degree of swell (as caused by the ettringite precursor) from being identified. Thus, the use of a swell test which adequately represents site conditions, especially temperature, is proposed as a fundamental requirement in defining the degree of risk from sulphate swell.

### 7.2 IMPLICATIONS FOR THE SPONSOR AND WIDER INDUSTRY

The majority of the findings apply equally to the sponsors and the wider industry. It would be very difficult for a single company to propose changes to a (British) standard swell test without the support of industry working groups. For this reason substantial effort to increase the study impact with the wider industry was applied (see Section 7.3).

The study has suggested that different types of lime binder should be used depending on the intended outcome of treatment. A major advantage with the use of quicklime is the very rapid water conditioning of an excessively wet fill material. However, this study identifies that a high degree of inter-clod porosity may develop if regions within the treated material becomes too strong and resist compactive effort. This increased susceptibility to water ingress may cause durability issues, which will compound if the treatment involves a high TPS clay.
A specific site relevant risk when using quicklime is foreseeable in a scenario where SLS treatment is applied to a wide area, for example a 300mm capping or sub-base replacement layer involving high TPS soil beneath the concrete slab for a large warehouse. If a quicklime + GGBS mix were planned, the wide area of the proposed treatment would require that overlapping treatment bays are used to mix the quicklime binder (see section 1.3.4). Where the treatment bays overlap, there will be a ‘double dose’ of quicklime binder. As noted in Section 1.3.4, this may cause up to 40% of the material to receive twice the amount of quicklime. So even if additional mixing water is added, the water condition of 40% of the material will be different (drier) to the remaining 60%. So post compaction, there is potential for this higher permeability to cause surface / ground water to easily soak into the treated material, directly increasing the risk of sulphate swell. Additionally, both the M40 and Wadesmill bypass failures used quicklime during periods of drought and high ambient temperature (Section 1.6.1). The heat from quicklime slaking would have compounded the drying influence of the weather. Thus a high degree of inter-clod porosity, especially at points of treatment overlap may have been a key factor in these failures. The use of hydrated lime binder under such conditions would lessen the potential for a high degree of inter-clod porosity to develop.

The study findings relating to sulphate swell working methodologies e.g. mellowing periods and GGBS partial replacement for lime are in general alignment with existing recommendations (Britpave 2005). However, the findings regarding hydrated lime are recommended for consideration.

Findings identifying a greater degree of sulphate swell at lower temperatures (8°C) are of specific importance in developing sponsor company and wider industry understanding of the risks associated with treating high TPS material. As the cure temperature and length of unsoaked cure period have potential to substantially influence the degree of swell determined
by any laboratory test, it is recommended the study findings and proposed further research are reviewed.

With regard to the suitability of sulphate swell tests, it is suggested that a test method allowing water to access all sides of a confined specimen is developed. A potential benefit from a test with these feature would be the scope to predict final swell from early readings, an aspect of considerable value when a slow rate sulphate swell continues for a very long time (see further research section 7.4).

7.3  RESEARCH IMPACT ON WIDER INDUSTRY

Throughout the EngD study period, considerable effort has been applied to consider relevant research and industry communities beyond the immediate supervision team. The motivation for this was:

- to ensure study focus aligned with wider industry requirements;
- to benefit from a wider area of knowledge;
- to enhance the impact of the study.

These efforts are summarised in Table 7.1, and while all have improved the study impact or focus in some manner, the most value has been added through engagement with the Britpave soil stabilisation task group (BSSTG) and a summary of this involvement is provided in section 7.3.1.

7.3.1  BRITPAVE SOIL STABILISATION TASK GROUP

Britpave is an independent UK organisation, with members including consultants, engineers, specialist plant and product suppliers, which work towards increasing the use of cementitious solutions for construction. The SSTG are a subgroup of the organisation specialising in soil
<table>
<thead>
<tr>
<th>Activity date / Purpose</th>
<th>External group / company</th>
<th>Description</th>
<th>Follow up activity</th>
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<tbody>
<tr>
<td>23rd March 2011 (Year 1) Raise EngD awareness</td>
<td>Geological society - Yorkshire group. (monthly meeting)</td>
<td>60 minute presentation. “Lime stabilisation – black art or geoscience”. Focus on literature explaining science underlying ameliorative and deleterious processes in SLS</td>
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<tr>
<td>10th January 2012 (Year 2) Raise EngD awareness; Industry feedback on early findings / forward approach</td>
<td>Lhoist Group (Major lime producer)</td>
<td>Meeting with Research and Development manager for Lhoist to present and discuss early nucleation trial findings and forward research direction.</td>
<td>Visit (30th May 2012) to Lhoist technical centre in Belgium to present/discuss Paper 2 with Lhoist soil stabilisation research team.</td>
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<tr>
<td>19th April 2012 (Year 3) Raise EngD awareness: - Paper 2 and 3</td>
<td>Geological society – East Midlands group (monthly meeting)</td>
<td>60 minute presentation. “Nucleation in lime stabilised soils”. Presentation of nucleation trial findings</td>
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<tr>
<td>31st May to 1st June 2012 (Year 3) Raise EngD awareness: - Paper 2 and 3</td>
<td>International Symposium on Ground Improvement (Brussels, Belgium)</td>
<td>Podium presentation (10 minute) of paper 2. Discussion of EngD with researchers from Newcastle and Cambridge Universities.</td>
<td>Visit to Newcastle University (21st September 2012) to discuss with lime stabilisation research group</td>
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<tr>
<td>25th July 2012 Industry feedback on sulphate swell trial proposals</td>
<td>BSSTG – sub group working on comparison of sulphate swell tests</td>
<td>Discussion of ongoing BSSTG work into comparing the results from 3 sulphate swell tests @ 20°C. BSSTG very interested in the NST proposals (see section 7.3.1).</td>
<td>Led to invitation to regularly attend BSSTG technical meetings</td>
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<tr>
<td>28th November 2012 Raise EngD awareness; Involvement with HA74 review</td>
<td>BSSTG technical meetings (quarterly)</td>
<td>Attendance at BSSTG meetings to discuss inclusion of EngD findings in HA74 revision. See section 7.3.1 for further details. A 30 minute presentation of the sulphate swell trial findings was delivered during the 29th April 2014 meeting (see section 7.3.1).</td>
<td>Regular attendance at subsequent quarterly meetings</td>
</tr>
<tr>
<td>26th September 2013 Raise EngD awareness - Paper 1 Involvement with HA74 review</td>
<td>Britpave – annual meeting and seminar</td>
<td>25 minute presentation “Developing soil stabilisation practice with industry focussed research”. Extended research findings from paper 1 to wider Britpave membership.</td>
<td></td>
</tr>
</tbody>
</table>

Table 7.1 Summary of engagement with wider industry and academic groups over EngD
stabilisation and are notable in their efforts to produce best working practice documents e.g. Britpave (2005).

Involvement of the EngD study with the BSSTG commenced in July 2012, when discussions related to the groups work on a comparison of results from three sulphate swell test methods (i.e. Higgins et al, 2013). The discussions were very useful in obtaining industry opinion on how to progress the sulphate swell trial. The BSSTG were particularly interested in the proposed inclusion of a temperature reduction, so the NST proposals were refined in conjunction with this dialogue.

Following this initial discussion, regular attendance at the BSSTG quarterly meetings was undertaken, which along with an increased awareness of the EngD study, led to three significant events:

- Presentation of key findings from Paper 1 at Britpave’s annual meeting and seminar;
- Presentation to the BSSTG of the implications of a temperature reduction on sulphate swell readings during the sulphate swell trial. An early introduction of this important finding to the wider industry;
- Involvement with a forthcoming revision to the HA74 guidance (see 7.3.1.1)

7.3.1.1 HA74 review process

The BSSTG were offered the opportunity to propose some changes to the key guidance document HA74 (The Highways Agency 2007). It is not clear how many or when the recommendations will be incorporated into the document, however, this provided a useful discussion forum for explaining the EngD findings to the BSSTG. The key points made during discussions of the revision are as follows:
• In the present document there is no differentiation between the chemical / physical processes driving lime only stabilisation, lime and cement stabilisation and cement only stabilisation. The document could be revised to set out how different binders cause strength improvement in different locations, i.e., lime stabilisation instigates diffuse cementation within clods and cement stabilisation binds clods together, lime and cement stabilisation may combine improvements from both diffuse and inter-clod cementation.

• The guidance highlights the need to use mandatory mellowing periods of 24-72 hours – as required by the MCDHW. As set out in Paper 1, mellowing with quicklime powder has potential to cause increased air voids from inter-clod porosity. The requirement to mellow should be determined from project specific factors and testing, not prescription and a technical discussion of the influences mellowing can cause would assist with this decision making process.

• The section on sulphate swell may be updated to include a discussion on recent work to summarise the possible strategies for reducing sulphate swell risk (i.e. as discussed in section 6.1.1-6.1.4). Thereafter, the issues relating to an increased rate of sulphate swell at site relevant temperatures when compared with those applicable at 20°C (as discussed in section 6.5.5 of this EngD) could be raised.

7.4 RECOMMENDATIONS FOR FURTHER RESEARCH

Significant areas requiring further research relate to:

• The site relevant curing regime for sulphate swell tests. The use of an 8°C water temperature in lieu of 20°C waster in soaked curing causes a greater degree of swelling to occur. Furthermore, the duration and temperature of the unsoaked curing period has potential to promote / prevent the growth of non-expansive ettringite prior to immersion. This requires a deeper consideration of the chemistry leading to sulphate swell and
ettringite precipitation at site temperatures (<10°C) and how a laboratory test curing regime may replicate this.

- Further research into a suitable swell test should be undertaken. The NST proposed here highlights the benefit of allowing water access to all sides of the specimen, while still applying confinement. A key benefit of the test is the high rate of early swell and the potential for the inversely proportional reduction in swell rate to predict the final swell from these early values.

- The ‘as planned’ further and repeat TGA and XRD analysis should be undertaken to improve the rigour of this work.

- The applicability of sulphate swell trial findings should for investigated in different natural clay soils. Of particular interest is whether the proposed methodologies to reduce risk are relevant to clays with a high pyritic content i.e. the pyritic Lower Lias clay.

- A satisfactory explanation for the increased swell behaviour in the CG_G_72 specimen at 8°C could not be given. Research into lime/GGBS stabilisation at low temperatures should investigate this further.

### 7.5 CRITICAL EVALUATION OF THE RESEARCH

The research has investigated site relevant processes using laboratory test methods, however, the actual site relevance has not been established through a comparison with samples obtained from a site trial. This appears particularly relevant for the sulphate swelling issues, where site temperature is considered a trigger for swell. This process would ideally be undertaken to complete this laboratory / site link and without it the site relevance of any sulphate swell test could be considered ambiguous.
A challenge towards the sulphate swell trial could relate to the lack of repeat testing for any individual sub batch. This may be particularly relevant when considering the NST was a new test method and a more robust approach would have trialled repeat specimens against a lesser number of variables. However, the use of the soaked CBR test method alongside, which showed very similar trends to the NST does help validate the NST. The same repeatability criticism can be applied against the XRD and DTA tests, however, it is planned to undertake repeat tests before publishing this work.

It was established during literature review that the most problematic material for sulphate swell was the pyritic Lias clay. However, the sulphate swell trial did not use this material and instead draws conclusions relating to temperature effects from gypsum rich clays. This is a potential limitation, however, it is known from literature that pyrite oxidation to gypsum is substantially accelerated during prolonged mellowing periods (Floyd et al. 2003). Thus, the proposed methodology should still reduce risk of sulphate swell in pyritic material, however, the extra chemical stage may require an even longer mellowing period? Further research has been recommended to cover this point.
8 REFERENCES


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Enhancing the understanding of lime stabilisation processes


http://www.britpave.org.uk/FreePublicationsSoilStab.ink:


References


Enhancing the understanding of lime stabilisation processes


APPENDIX A  LIME STABILISATION FOR EARTHWORKS:  
A UK PERSPECTIVE (PAPER 1)

Full Reference


Abstract

Lime stabilisation is a versatile technique applied during earthworks operations. Modern soil recycling units are much more efficient at pulverising fill material and inter-mixing the added binder / water than machinery available 20 years ago. While supplier innovation adds flexibility to the site working method, specifications have not been sufficiently updated to permit optimal application. This review paper details the physico-chemical changes instigated through the lime-clay soil reaction, updating previous reviews. It aims to assist scientific debate, current practitioners and future specification changes. For example, the application of the minimum 24 hour mellowing periods (mandatory to UK specifications) with high reactivity, quicklime powders is concluded to cause increased air voids in the compacted fill. Increased air voids are associated with reduced long term strength and potential volume change from water ingress, which is of particular concern for sulphate swelling. Shorter mellowing periods and / or use of hydrated lime may lesson this issue, however, a ‘one size fits all’ approach is discouraged in preference to site specific methodologies refined to suit the fill material and project requirements. The paper discussion also summarises working methods which may lower the risk of sulphate swell and defines areas requiring further practical research.

Key words

Recycling & reuse of materials; Geotechnical engineering; Reviews
1 INTRODUCTION

The improvement of engineering properties of clay fills through the addition of a small percentage of lime binder has been a popular ground improvement technique in the UK since the 1970’s (Norman, 2011). Following the introduction of this technique, industry and academic efforts have been applied to better understand the mechanisms behind the observed ameliorative effects. This combined effort has advanced the state of knowledge and practice to identify three fundamental effects caused by reaction of lime with clay minerals, i.e. moisture conditioning, cation exchange and pozzolanic reaction (Sherwood, 1993). These processes generally realise a favourable outcome. However, heterogeneous soil chemistry and mineralogy may result in important changes to the nature of reactions between lime and soil. Practically, this prevents a ‘one size fits all’ approach to lime stabilisation and highlights the importance of a detailed understanding of the science underlying the lime-clay soil reactions for those involved in the practical application of this process. With this understanding, field processes can be changed according to the soil’s chemistry and mineralogy to ensure the desired outcome, i.e. an engineered material with a suite of appropriate characteristics that meet project requirements.

As sustainability drivers increase pressures to minimise landfill and re-use materials, research into the potential for incorporating waste derived pozzolans is increasing. For example, laboratory work has identified potential in incorporating wastepaper sludge ash (Rahmat and Kinuthia, 2011) and red gypsum (Hughes et al., 2011) into binder mix designs. These ‘new pozzolans’ add different types and ranges of chemistry/mineralogy. This imposes further layers of complexity on an already challenging topic and although the results are promising for the specific clay materials tested under laboratory conditions, the implication of soil heterogeneity relevant to site applications are less well understood.

It is timely, therefore, to summarise the state of science and practice, focusing on a simple case of lime stabilisation. Compiling this information into one point of reference provides substantial benefit for organisations engaged in soil stabilisation projects and also provides a point of comparison for any future work with new pozzolans. This paper reviews literature and applies experience of site practice to interpret the key aspects relevant to achieving desired performance in field applications of lime stabilisation. As this experience is predominantly from UK practice, comments relating to specification are based upon the Highways Agency specifications and guidance notes (i.e. The Highways Agency, 1995, 2007, 2009). However, while specifications may vary, the reasoning presented is relevant globally.

The paper is structured to first introduce the context of lime stabilisation and principles underlying lime-clay reactions (Section 2) before factors important in applying these principles at the field scale are described (Section 3). A discussion of how these concepts may promote a strong, durable and volumetrically stable fill material, including consideration of sulphur swell follows (section 4). Discrepancies between theory and practice are then summarised and
suggestions where enhanced knowledge would add value to practical applications are made (Section 5).

2 CONTEXT OF LIME STABILISATION

Lime is typically applied to clay soils that, in an untreated condition, provide problems for construction. In particular, shrink/swell of the substrate may damage engineering structures. Clay soils with a liquid limit ($W_L$) greater than 90% or a plasticity index ($I_P$) greater than 65% are thus classified as unsuitable for use as an earthworks fill material, unless treated (e.g. with lime; The Highways Agency, 2009).

Rapid improvements to the soil’s engineering properties are commonly referred to as “lime improvement” (separating it from ‘lime stabilisation’; Table 1) and have two facets. The first relates to conditioning of the fill to the Optimum Moisture Content (OMC) for compaction. The second relates to chemical alterations at the clay particles surface which decrease the volume change potential of the clay minerals.

<table>
<thead>
<tr>
<th>Treatment Intent</th>
<th>Physico-chemical Process</th>
<th>Common terminology</th>
<th>Indicative lime requirements</th>
<th>Typical time required*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lower the moisture content of a wet / low strength soil towards OMC. Either for compaction as a general fill, or to enhance trafficability.</td>
<td>1. Removal of free moisture by reaction with quicklime; 2. Cation exchange / clay mineral aggregation effectively increasing the OMC. (see 2.4.1 to 2.4.3 for detail)</td>
<td>Lime Improvement</td>
<td>Low, e.g. 0.5-4%. (initial moisture content / clay content dependent)</td>
<td>1. Immediate 2. Rapid (0-72 hours)</td>
</tr>
<tr>
<td>Reduced plasticity / potential for volume change.</td>
<td>3. Cation exchange / clay aggregation reduces clay mineral effective surface area and affinity for water. 4. Early pozzolanic reactions restrict subsequent dispersion of aggregations (see 2.4.3 for detail)</td>
<td>Lime Improvement</td>
<td>In excess of the ICL value, e.g. 2 – 10%; (actual binder addition determined by site specific mix design)</td>
<td>Rapid (0 – 72 hours)</td>
</tr>
<tr>
<td>Substantially improved engineering properties i.e. strength, stiffness and durability.</td>
<td>5. Pozzolanic reaction between lime-clay soil system. (see 2.4.4 for detail)</td>
<td>Lime Stabilisation</td>
<td>On-going improvement from 72 hours continuing for months / years</td>
<td></td>
</tr>
</tbody>
</table>

* Indicative times only. Actual time depends upon multiple factors detailed in later text.

Table 1. The treatment intent and the implications for lime addition and time dependent reactions.
With greater additions of lime binder (i.e., in excess of the initial consumption of lime (ICL) value; BSI, 1990) pozzolanic reactions between lime and clay can result in substantial long-term enhancement of engineering properties, including high strength, durability and frost resistance (Eades and Grim, 1966). The added benefits from the pozzolanic reactions are experienced more slowly than the rapid lime improvement reactions, and can progress over months and even years. Lime may also be used in combination with additional binders, for example Portland cement, or through combined reaction with pozzolans such as ground granulated blast furnace slag (GGBS). Such approaches may be sought when greater strengths are required rapidly (e.g. within 7 days). It is important to note that these approaches alter the reaction product chemistry and locations where strength improvement takes place, when compared to soil treatments involving lime only (Wilkinson et al., 2010).

2.1 Volume Stability

The volume stability and durability of a lime treated fill is of paramount importance. Although the majority of soil stabilisation works result in the desired performance (Petry and Little, 2002), occasionally problems occur. Following high profile failures in the United Kingdom (M40 motorway, Oxfordshire; Snedker, 1996) and United States (Las Vegas; Hunter, 1988), the potentially deleterious reaction between sulphur and a lime-clay soil system has received substantial attention. There is a considerable body of work supporting the notion that this combination of reactants may lead to the precipitation of ettringite and/or thaumasite (Little et al., 2005). The formation of ettringite, in particular, may be accompanied by substantial expansion with the potential to cause damage to engineering structures (Snedker, 1996).

The potential to generate problems for construction due to adverse soil chemistry is very much smaller than the potential to develop problems due to a high air voids percentage of the lime treated soils. A major factor in addressing the latter involves the early lime-clay soil reactions in combination with the working methodologies employed and this paper addresses this in some detail below. A potential for sulphur swell may be broadly defined from the association of sulphur species with certain geological formations (Figure 7). However, as sulphur swell issues from soil stabilisation are relatively rare and material from these potentially sulphur bearing formations (Figure 7) have also been successfully treated, it becomes apparent that the issue is more complex than this general association and section 4.3 expands considerably on this point. Notwithstanding, industry experience suggests that sulphate swell potential is of specific concern with high sulphur clay soils that are also of high plasticity, with the Lias Clay Formation having notoriety in this regard (Snedker, 1996; Holt et al., 2000).

2.2 Role of water

A particular amount of water is required to achieve an OMC that coincides with the maximum dry density (MDD). In addition, the initial moisture content dramatically influences the manner that lime diffuses through and reacts with the clay system. A greater initial moisture content enhances the efficiency of lime diffusion (Barker et al., 2007) and provides sufficient water to precipitate pozzolanic hydrates in the longer term (Bell, 1988). This illustrates the importance
of understanding the intent of the lime treatment. A focus on achieving MDD for bulk fill compaction does not fit well with achieving efficient development of the long term pozzolanic reactions and vice versa. Furthermore, the timing of water availability is critical in determining whether ettringite growth results in soil expansion (Little et al., 2005). Therefore, the role of water management within a lime-soil system receives specific attention in this paper.

2.3 Lime types
Lime binder may refer to quicklime (CaO) or hydrated lime (Ca(OH)$_2$) and may be provided in various forms, i.e., pellets, flakes, powder or as particles suspended within a water slurry. Each variant of lime binder is produced to different physical / chemical criteria (e.g. as categorised in BSI, 2006) and may suit a specific soil stabilisation application better than another type/form. Lime type is considered further in section 3.4. However, the sequence of lime-clay reaction must first be discussed, before implications of lime binder type can be considered.

2.4 Lime–clay soil reactions
This initial discussion of the underlying reactions assumes the added lime is in close contact with the reacting clay component and ignores any requirement for the lime constituents to first migrate into position. The latter complication is added by field applications of lime stabilisation which process larger clods of clay and will be considered in section 3.2. The processes are discussed in the order in which they are considered to occur in a field scenario which aids later correlations to physical effects.

2.4.1 Drying
Substantial drying is only applicable where quicklime is used as the binder, removing excess water from the soil system through the action of “slaking” the quicklime (CaO) to calcium hydroxide (Ca(OH)$_2$). This combines 32% of the quicklime’s initial mass with water molecules, causes an increase in volume (potentially providing deleterious expansive stresses if in a confined environment). The resultant exo-thermic reactions generate substantial heat. This heat causes evaporation which further reduces the moisture content (Greaves, 1996). Therefore, the influence is principally one of drying out the material.

2.4.2 Calcium hydroxide dissociation
Whether added directly, or produced through quicklime slaking, calcium hydroxides enter the soil water solution. Calcium hydroxide is relatively stable in water, although it can partially dissociate to provide calcium (Ca$^{2+}$) ions and hydroxyl groups, which may then react with the clay-soil system (Bergado, 1996). The hydroxyl groups also elevate the pore water pH to a maximum value of approximately 12.45. Calcium hydroxide dissociation is a pre-requisite to subsequent changes that determine the engineering properties of lime-clay mixes.

2.4.3 Cation exchange and soil structure change
The modification of the clay structure mainly involves the calcium ions and is regarded as a rapid cation exchange process occurring on the surface of clay particles. Clay particles typically
exhibit surface charge imbalances and the negative charges are balanced by hydrated cations. According to Van Olphen (1977), the thickness of the diffuse double layer is controlled by several factors, one of which is the hydration radius of the charge balancing cations (Reeves et al., 2006). Hence, incoming divalent calcium cations, of a smaller hydration radius, exert a greater attractive force towards the clay particle surface than any monovalent cations (which are common to natural clay soils, e.g. K⁺ or Na⁺) and the thickness of the diffuse double layer shrinks in response (Bohn, 2002). As the diffuse double layers shrink, the electro-static charges on adjacent clay particles interact to a greater extent. Opposing negative charges of parallel aligned (face to face) clay particles are repelled and reconfigure to promote a flocculated, positive / negative charge (e.g. edge to face) arrangement (Figure 1). This causes silt sized aggregations of clay particles to group together (Bell, 1996) and two influences on the clay soil structure are suggested; an increase in micro-porosity, intra-aggregate to the flocculated particles (Figure 1c); and a change to the meso-porosity, inter-aggregate to the flocculated particles (Figure 1d). This reduces the effective surface area of clay minerals in contact with the inter-aggregate pore water accounting for much of the immediate change in physical properties of the clay soil associated with lime improvement (Figure 2; Bell, 1988):

- Reduced plasticity and shrink / swell;
- Promotion of brittle / friable behaviour;
- Increased permeability.

**Figure 1** Sequence illustrating influence of early lime-clay reactions upon clay particle arrangements and soil structure
As the described change in soil structure is dependent upon a suppressed diffuse double layer it might be expected that the effects could be reversed through suspension within water. However, this is not evident and the aggregations persist, even when reworked during classification tests, e.g. particle size distribution and Atterberg tests. This resilience is caused by the rapid growth of some pozzolanic reaction products (Diamond and Kinter, 1965). These early formed reaction products localise around points of contact (e.g. edge / face) between clay particles within the flocculated structure (Figure 1c). This discrete strengthening at point of contact explains how formation of a very small quantity of reaction product provides resilience against dispersion (Diamond and Kinter, 1965).

2.4.4 Pozzolanic reactions

Pozzolanic reactions may be broadly summarised as new mineral growth, facilitated by a high pH pore water (i.e. pH >9) and involving reactants sourced from either the added binder, host clay soil, or transported in from an external source, i.e., solutes within the pore water (Diamond and Kinter, 1965; Sherwood, 1993; Bell, 1996; Boardman et al., 2001). With the noted exception of early formed reaction products (Diamond and Kinter, 1965), long curing periods (weeks and months) are required for the newly formed minerals to provide notable and ongoing benefit. The increase in strength with curing is attributed to the new minerals binding adjacent aggregations of clay (Figure 3; Bell, 1996). Wild et al., (1987) suggested that by in-filling the inter-aggregate pore space, strength increase and permeability reduction would also occur through ‘pore blocking’.

Clay soils are typically rich in alumino-silicates and reaction products similar to those produced through hydration of Portland cement form, e.g. calcium silicate hydrates (C-S-H), calcium aluminate hydrates C-A-H and calcium aluminate silicate hydrates (C-A-S-H). The composition of the reaction products formed by the lime-clay soil reaction is necessarily driven by the timing of reactant availability. The abundant early supply of an alternative chemistry, e.g. $SO_4^{2-}$ ions, in lieu of silicate, may dramatically change the reaction product from an...
ameliorative into a potentially deleterious mineralogy. This introduces the concept of reactant timing and shall receive further attention in sections on ion migration at the field scale and adverse chemistry (see 3.2 and 4.3).

Figure 3  Environmental scanning electron microscope images of lime treated clay (32 day cure) illustrating pore water / C-S-H gel connecting individual aggregations with little visible air voids. (The image in figure 3a is reproduced with permission of the Transportation Research Board; Beetham et al., 2014).

3 TRANSFERRING SCIENCE TO PRACTICE

3.1 Clay clods

Up to this point, much of the discussion involved processes operating at the microscopic scale and assuming an intimate lime-clay mixture. This can make it difficult to relate to site processes where the rotovating / mixing action of site machinery tends to produce clods of clay soil (which may be up to 50 mm in diameter) with lime applied to the periphery of these clods. Lime stabilisation has some aspects similar to standard earthworks, e.g., the immediate strength is mostly influenced by dry density. However, the inclusion of lime binder in conjunction with the clod size adds a further strength dynamic. Upon immediate contact with lime, the clay clods go through the lime–clay reaction sequence as described above and this alters the fill properties in an on-going manner. The lime is initially localised along the periphery of the clods and for the lime-clay reactions to extend beyond the surface of a clod, the calcium ions and hydroxyl groups must transport deep into the clod.

3.2 Lime migration

Ion transport in clay soils is primarily driven by diffusion along electro-chemical gradients where ions redistribute to regions of low concentration (Jungnickel et al., 2004). Although the diffusion process dominates in soils of high plasticity, advection becomes increasingly influential with increased permeability, i.e. with permeability greater than $1 \times 10^{-9} \text{ m/s}$ (Mitchell and Soga, 2005). Therefore, where soil plasticity is low or there is a significant granular component, advection may become the key ion transport mechanism. While ion diffusion
predominantly involves the migration of charge balanced anion/cation pairs (Jungnickel et al., 2004), in a lime treated clay soil, the distribution of Ca\(^{2+}\) occurs more rapidly and with wider coverage than the hydroxyl groups (Davidson et al. 1965). This suggests that lime does not diffuse as paired Ca[OH]\(_2\) as might be anticipated in an advective system. Rogers and Glendinning (1996) attributed the difference in migration distance and rate to the high reactivity of OH\(^-\) with the surface of alumino-silicates. However, in a saturated soil condition connected pore water enhances thermal conductivity to stimulate molecular excitement, and this substantially accelerates the rate and extent of OH\(^-\) diffusion (Barker et al., 2007). Beetham et al. (2014) reported that when clods of clay were at a high degree of saturation before lime treatment, the elevation of pore water pH to more than 10 throughout 20 mm clay clods is achievable within several days. They also noted that the reduced surface area of clay particles, flocculated by the preceding Ca\(^{2+}\) cation exchange process, would also decrease OH\(^-\) interactions, promoting the wider migration of these hydroxyl groups.

Following movement of Ca\(^{2+}\) ions and hydroxyl groups into place, the subsequent development of pozzolanic reactions throughout the clods is given the distinction ‘diffuse cementation’ (Stocker, 1975). As the pozzolanic reaction mechanism relies upon the connectivity of pore water around clay aggregations, the potential for these reactions to extend over the larger, air filled inter-clod porosity is limited. Therefore, while lime stabilisation has potential to develop substantial diffuse cementation, unless other binders are added (such as Portland Cement or alkaline activated GGBS) there is little potential for ‘inter-clod cementation’. It has not been universally accepted that diffuse cementation plays a significant role within lime stabilisation. Some authors have suggested that ion diffusion can only occur to a very limited degree (e.g. Ingles and Metcalf, 1972; Petry and Wohlgemuth, 1988). This may be due to the experimental conditions that led to these conclusions either being sub optimal for efficient ion migration (i.e. unsaturated soil condition), or promoting another influence upon strength to occur more rapidly, thus causing diffuse cementation to appear insignificant. With the former, there are multiple influences on the rate of pozzolanic reaction to consider and this will be explained in section 4.1.2).

3.3 Compaction

The principles of compaction for a lime improved or stabilised clay are similar to those influencing a standard clay soil improved by mechanical compaction. In general, MDD corresponds with the maximum shear strength (attainable immediately). However, with the addition of lime, the MDD attainable from a treated fill reduces and the OMC typically shifts towards the wet side (Figure 4; Bell, 1996). Lime treatment tends to produce a relatively flat curve when results of dry density versus moisture content are plotted, which in some situations may flatten completely so that a lesser or no increase in MDD is associated with an OMC.
3.3.1 **Inter-clod porosity**

The reduction in MDD has been linked with the flocculation of clay particles causing an increase of porosity and a lower density (Sherwood, 1993). However, as the size of pore space imposed by clay particle flocculation is considered to be <0.3µm (according to Bin et al. 2007), it is unlikely that this intra-aggregate porosity could fully account for the larger reductions in dry density. In a study on compacted clods of untreated clay of high plasticity, Benson (1990) associated large reductions in dry density and increased permeability with increased void space between clay clods (i.e. an inter-clod porosity with pore sizes >100µm); influences from changes to micro-porosity (i.e. pore spaces <1µm) were minor. The key influences on this inter-clod porosity are clod ductility, clod size and compaction effort (Benson, 1990). Sweeney et al., (1988) identified how both increased quicklime content and mellowing period (from 1 to 24 hours) promote the early lime clay reactions that reduce the ductility of clay soil. With a shorter mellowing period and lower amounts of added quicklime the modified Proctor (4.5 kg) compaction results showed an increase of the MDD at OMC, while a flat compaction curve resulted with normal Proctor (2.5 kg) compaction. However, for samples subjected to longer mellowing periods, a much less ductile material resulted and the higher compaction energy did not realise an increase in MDD. Instead a weak and friable specimen, prone to breaking up, was produced (Sweeney et al., 1988). Other authors have similarly noted that increased mellowing periods promote a substantial increase in the percentage of air voids (Bell, 1988; Holt et al., 2000). The early lime-clay reactions introduce a brittle response and moisture conditioning to the wet of OMC will have only limited effects. Further compactive effort may result in additional voids by fracturing brittle clods and inducing dilatancy.
3.4 **Clod size / strength heterogeneity**

The initial clod size of treated clays plays an important role in the timing of lime-clay reactions and the potential for inter-clod porosity. While compaction of (untreated) smaller clods reduces inter-clod porosity (Benson, 1990), the lower volume of clay over which ion diffusion must progress causes lime-clay reactions to develop throughout small clods (e.g. 5 mm or smaller) at a faster rate than for larger clods (e.g. 20 mm or larger; Petry and Wohlgemuth, 1988; Bozbey and Garaisayev, 2010; Beetham *et al.*, 2014). The rotovating action of site plant produces a range of clod sizes, thus the rate of change in clod ductility/strength will be heterogeneous, i.e. fast for the small clods, slow for the larger clods. When the range in clod size is within a reasonable banding before lime coating (i.e., as attained by the initial rotovation only), this should not prove a major issue with regard to compaction timing. However, the use of quicklime, particularly in combination with initially large clods has high potential to exaggerate this heterogeneity. Beetham *et al.* (2014) reported on the effects of a sequence comprising the addition of quicklime followed by a 1-hour mellowing period and then remixing before compaction. They observed that this caused fine particles to break away from clods (up to 20 mm size) of a moderately highly plastic clay. As these fines originated from the clod periphery, these contained a disproportionately high concentration of lime, which is then isolated within the fines and away from the larger clods. This effect further exaggerates the heterogeneous development of early lime-clay reactions (Figure 5). It is also logical to suggest that the use of quicklime powder would worsen this effect. The rapid liberation of heat when category 1 quicklime (BSI, 2006) slakes would encourage both a localised drying and an acceleration of

![Figure 5](image.png)

*Figure 5* Sequence illustrating the influence of quicklime slaking upon larger clay clods to produce a heterogeneous distribution of lime (after Beetham *et al.*, 2014)
lime-clay reactions. As diffusion is optimal with a saturated clay soil, migration to the centre of these clods would then be restricted and a drying, brittle rind would rapidly form leaving a core zone where relatively little alteration takes place (Figures 5c & 5d). Therefore, unless the remixing phase is accompanied with further binder addition the large remnant cores may receive very little lime.

3.5  Working method influences

To limit heterogeneous distribution of lime and moisture, the mellowing period may be extended to allow the lime improvement processes to substantially complete throughout clay clods. The lime improvement process is usually complete within 24 hours, but subject to clay mineralogy and content may require up to 72 hours (Rogers and Glendinning, 1996). Large clods may require even further time. Encouraging heterogeneity with a prolonged mellowing process would promote the high air voids issue previously identified, especially when quicklime powder is used. This suggests that, unless the application of quicklime can realise the pulverisation specification within a relatively short mellowing period either high air voids percentage or a heterogeneous mix will result. To avoid heterogeneity and minimise air voids where larger clods persist, the use of hydrated lime powder in lieu of quicklime is preferable. This would avoid the complication of a drying rind (Figure 5) and permit longer mellowing periods before ductility is lost. A further option, involves the use of hydrated lime/slurry and to deliberately target a substantially wet of OMC condition, perhaps with minimal mellowing. The principal issue here is to accept a reduced immediate strength, and rely on optimising diffuse cementation processes to achieve a higher strength as curing continues to take place (Figure 6a & 6b). This may be most suited to highly plastic clays where pulverisation is inherently difficult and the clay mineralogy promotes diffuse cementation in a more expedient manner.

It becomes apparent that different combinations of moisture conditioning, mellowing periods and lime binder type will suit different clay materials and the key consideration to achieve the optimal approach is the performance requirements of the compacted fill. The performance element should consider the medium (31 days) and long term (more than 6 months), which may not be indicated by immediate or short term (7 day) testing and would instead require an in depth understanding of the principals behind durability (section 4.2) and cured strength gain (section 4.1.2). It is worth repeating here that the OMC for immediate strength does not necessarily coincide with the moisture content required for maximum long term strength and durability. Providing that sufficient strength is gained to permit immediate construction requirements, it may be preferable for some applications to target higher moisture.
4 CURED STRENGTH AND DURABILITY

4.1 Pozzolanic reaction and reactant timing

With respect to a detailed micro-chemical explanation for the pozzolanic reactions, it is important to acknowledge that this is a complex subject and the present state of science has not achieved complete agreement. For this reason, a complete discussion would require a separate review paper and a detailed summary is not attempted here. Instead, it is noted that of the different reaction mechanisms suggested in the literature, they may generally be grouped into those involving the topo-chemical alteration of an alumino-silicate host to form the new phase in situ (e.g., Stocker, 1975); or via a through solution processes, i.e. the dissolution of reactants from an alumino-silicate host, followed by transportation and then precipitation of the new phase at an alternative location (e.g., Beetham et al., 2014). While the topo-chemical reactions would explain surface changes, such as Diamond and Kinter’s (1965) early pozzolanic alteration, the on-going growth of reaction products into the inter-aggregate pore space (Wilkinson et al., 2010; Wild et al., 1987) suggests that a ‘through-solution’ mechanism is necessary for any further pozzolanic reaction to take place beyond initial contact amelioration. It has already been identified that the efficient distribution of hydroxyl groups is driven by a connected pore water and this would be pre-requisite to the initiation of both topo-chemical and through-solution processes.

4.1.1 Through Solution Mechanism

When the pore water alkalinity exceeds a pH of 9 it generates a corrosive environment in which the alkaline hydrolysis of covalent bonds between Al-O and Si-O release monomeric silicate and aluminates (e.g. SiO$_4^{4-}$ and Al[OH]$_4^{-}$) into solution (Cristelo et al., 2012). Water forms an essential component to enable this reaction and further highlights the importance of a connected pore water for the sustained supply of reactants. With regard to the supply rate of these reactants there are three direct influences:

- the availability of alumino-silicates;
- the strength of the covalent bonds in the alumino-silicate; and
- the corrosivity of the pore water.

Additives comprising bases stronger than Ca(OH)$_2$ have been shown to enhance alumino-silicate dissolution (Diamond and Kinter, 1965), with NaOH and KOH providing a pH of 13.5 to 14 and substantially accelerating this attack (Elert et al., 2008, Duxson et al. 2007). The availability of alumino-silicates is essential to pozzolanic reactions, although the quantity of dissolved silicate and / or aluminates required to sustain the reaction is small and once an initial minimum is met (e.g. plasticity index more than 10) the clay content is not limiting (Bell, 1988). The rate at which these reactions result in an increase in strength is linked to an hierarchy of clay minerals where expansive clay minerals, such as montmorillonite, are recognised as providing the greatest rate of dissolution thus enabling maximum efficiency of pozzolanic reactions. The high reactivity of these expansive clay minerals is determined, among others,
by their very high surface area and the extra silicate layer within 2:1 minerals (Table 2; Elert et al. 2008, Ingles and Metcalf, 1972). These cannot be the sole factors, because Ingles and Metcalf (1972) consider the reactivity of illite to be less than that of kaolinite. In this regard, Bell (1988) suggested that not all the silica in 2:1 clay minerals would be freely available, with illite and chlorite as examples where ionic bonding might restrict silica availability. Additionally, Al-O covalent bonds are weaker than Si-O bonds and therefore aluminate supply will initially exceed silicate supply. However, as clay soils have a greater overall silica content, this supply rate will gradually reverse with on-going dissolution (Cristelo et al., 2012). The degree of crystallinity within the alumino-silicate source also has a high influence on bond strength and compared to well-ordered clay minerals, amorphous alumino-silicates rapidly dissolve (Duxson et al., 2007). This also explains why some industrial processed alumino-silicates, which have been subject to prior significant heat (e.g. GGBS, meta-kaolin), have little order and are rapidly activated by calcium hydroxides (Cristelo et al., 2012).

<table>
<thead>
<tr>
<th>Clay Mineral</th>
<th>Layer structure</th>
<th>Surface area [m²/g]</th>
<th>Swelling properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Kaolinite</td>
<td>1:1</td>
<td>10</td>
<td>None</td>
</tr>
<tr>
<td>Allophane</td>
<td>1:1</td>
<td>700-900</td>
<td>None</td>
</tr>
<tr>
<td>Smectite</td>
<td>2:1</td>
<td>800</td>
<td>Extensive</td>
</tr>
<tr>
<td>Illite</td>
<td>2:1</td>
<td>20</td>
<td>None / very little</td>
</tr>
<tr>
<td>Vermiculite</td>
<td>2:1</td>
<td>400</td>
<td>Limited</td>
</tr>
<tr>
<td>Chlorite</td>
<td>2:2</td>
<td>10</td>
<td>None</td>
</tr>
</tbody>
</table>

Table 2 Basic properties of common clay minerals (after Rowell, 1994)

The newly formed minerals are produced from solutes released into the pore water. An initial Al/Si complex of high reactivity and mobility will form and its subsequent evolution is highly dependent upon the supply of other ions (Cristelo et al., 2012). Where calcium is available, the rapid formation of C-S-H gel takes preference, although the composition of the reaction product will depend upon the concentration of ions and local energetics (Duxson et al., 2007). A connected pore water environment would assist in the promotion of a more homogenous reaction product. Locally greater concentrations of aluminate may result in precipitation of poorly crystalline C-A-H or C-A-S-H phases. This is an inherently meta-stable system and phase changes may continue as ion supply alters, although as the solid component of the gel increases, re-organisation becomes progressively high energy and a greater resistance to dissociation develops (Duxson et al., 2007). Furthermore, as the newly formed minerals grow within the inter-aggregate pore space, this progressive reduction in permeability (Wild et al. 1987; Beetham et al, 2014) would limit any new ion supply, further encouraging stability.

4.1.2 Strength gain rate

While the above sequence is an ideal progression, strength development and transition to a relatively stable, low permeability condition are dependent upon a number of factors that limit reaction rates, including:
Figure 6. Interpretative sketch (a) and plot (b) indicating how the pre-compaction condition of lime treated clay clods may influence the shear strength over time. When comparing a low water content system (an optimum moisture content for a maximum dry density) to a high water content system (efficient diffuse cementation), the latter will contain a period of time where the shear strength is less before it is recovered with curing. The duration of this period is influenced by the pozzolanic reaction rate (section 4.1.2). Inter-clod porosity which persists as a long term weakness is increased where clod strength is initially high and resists the compaction effort applied.
• initial ion diffusion efficiency – such as clay clod volume, degree of saturation, quantity of added lime;
• alumino-silicate reactivity – this is influenced by clay mineral type, and;
• other factors – such as curing temperature; pH-reducing organic matter or high sulphur content (section 4.3)

The time dependent strength influence of ion diffusion rate and alumino-silicate reactivity is indicated in Figure 6.

4.2 Stabilised soil durability

Durability of a lime-stabilised soil is judged against the sustained achievement of the required engineering properties. In view of the near-surface environment, changes caused by fluctuations in water content and temperature (including freeze-thaw cycles) are foreseeable events with the potential to impart changes upon the soil strength and volume stability.

The ingress of water has the potential to influence the residual clay mineralogy in a similar manner to a natural clay soil although, as previously noted, the low surface area of the resilient clay aggregations will lessen this influence. Thereafter, the stability of the pozzolanic reaction products becomes of interest. When submersed within water of neutral pH and low Ca\(^{2+}\), C-S-H gel will de-constitute into Ca(OH)\(_2\) and silicate (Taylor, 1990). The release of these components will then effectively raise the pH and Ca\(^{2+}\) of the water, and for this attack to be sustained, further water must then be supplied. This is supported by McAllister and Petry (1992) who identified that where permeability is high, the leaching of Ca\(^{2+}\) from a cured lime-clay system is sustained at a high level for an ongoing period. Conversely, for the low permeability system initial Ca\(^{2+}\) leaching was much lower and then stopped after only a short time period (Mcallister and Petry, 1992). Therefore, a high permeability material can potentially lose strength through softening and removal of the pozzolanic reaction products (Le Runigo et al., 2009).

Frost susceptibility is also primarily controlled by permeability, with the accumulation of water within the inter-clod void space (Figure 6a) comprising the main area where these expansive forces concentrate (Sherwood, 1992). Thus, the persistence of high air voids are directly associated with a reduction in durability and it is recommended that compaction achieves a minimum value of air voids throughout the fill (Sherwood, 1992).

4.3 Adverse Chemistry

In addition to the effects of poor compaction, various deleterious effects are associated with adverse chemistry. For example, heave and strength loss of lime and cement stabilised soils has been associated with high sulphur contents in the host soil, leading to the formation of the expansive and strength depleting minerals ettringite and thaumasite (Hunter, 1988; Snedker, 1996).
The formation of ettringite requires a pH greater than 10 and the adequate supply of chemical reactants; Ca$^{2+}$; SO$_4^{2-}$; Al$_2$O$_3$; H$_2$O to form:

$$\text{Ca}_6[\text{Al(OH)}_6]_2(\text{SO}_4)_3 \cdot 26\text{H}_2\text{O (ettringite)}$$

(Little et al. 2005)

Thaumasite may form in a similar fashion, or can generate upon alteration of ettringite (Little et al., 2005; Winter, 2009). A low temperature of between 4°C and 15°C and humid conditions are associated with thaumasite formation (Collett et al., 2004; Winter 2009). For the formation of thaumasite the following reactants combine; Ca$^{2+}$; Si$^{4+}$, SO$_4^{2-}$; CO$_2^{-3}$ and H$_2$O:

$$\text{Ca}_3[\text{Si(OH)}_6](\text{CO}_3)(\text{SO}_4) \cdot 12\text{H}_2\text{O (thaumasite)}$$

(Little et al, 2005)

Several strategies to prevent deleterious mineral growth, with a primary focus on ettringite, are described in the literature. These are listed below and discussed under appropriate sub headings:

1. Limiting / preventing access of one or more of the reactants;
2. Deliberate ettringite nucleation before final compaction;
3. Amending the chemical balance of a system to instead precipitate a non-deleterious mineralogy.

### 4.3.1 Limited reactants

It is apparent that sufficient quantities of calcium, aluminium, sulphate and water must be available to provide an environment in which ettringite can be formed. When soils are compacted to achieve a low air voids (i.e. less than 5%) this will drastically limit the potential for subsequent water ingress (Perry et al., 1996) and it will also limit the leaching of calcium ions from any prior formed C-S-H or portlandite (Mcallister and Petry, 1992). It is interesting to note that widely reported sulphate swell failures (for example Stewart Avenue, Las Vegas, US reported by Hunter (1988); and the M40, Oxford, UK reported by Snedker (1996) occurred when a quicklime binder was applied. Inter-clod porosity may have been a substantial factor in facilitating ettringite growth and subsequent swelling. Inter-clod porosity would be of further significance where it provides a pathway for ingress of sulphates from an external location.

### 4.3.2 Controlled ettringite growth

Ettringite is only expansive if the water is sourced from outside the immediate system i.e. post compaction (Little et al., 2010). Furthermore, where all reactants are immediately available, ettringite will form and reach a steady state within 150 hours (Little et al., 2005). However, the heterogeneous distribution of reactants, in combination with the low solubility of sulphate will further slow ettringite precipitation in a site application. If diffusion of reactants is encouraged through addition of sufficient water at the mixing stage (3-5% greater than the OMC) and this is coupled with an extended mellowing period (e.g. 3-7 days), this promotes the widespread nucleation and some early ettringite growth before final compaction takes place (Little et al., 2010; Petry and Little 2002). This strategy will limit expansion by encouraging initial ettringite growth before compaction, and then facilitating any subsequent expansive ettringite growth (as
sulphate solubility allows) over a greater area. The latter would promote the wider distribution of smaller ettringite crystals, so there is increased potential for this volume change to be accommodated by the inter-clod pore space; thus avoiding expansion hotspots (Little et al., 2010).

4.3.3 Chemical balance/timing

Increasing silica activity in a clay soil that has a chemistry that would normally form ettringite could instead lead to the formation of a volumetrically stable mineral, e.g. the calcium aluminosulpho silicate – prehnite (Little et al., 2005). Thus binder mix designs including products with rapidly available silica such as GGBS have been linked with the prevention of sulphate heave (Higgins, 2005). As an alternative hypothesis to how GGBS restricts sulphate heave, Wild et al. (1996) suggested that the GGBS particles, which also contain aluminium, provide an alternative surface for ettringite nucleation. They proposed that ettringite growth extending from the surfaces of GGBS particles was non-expansive, whereas if ettringite growth nucleated from the edge site of a clay mineral this was associated with expansion (Wild et al., 1996). It may be that the non-expansive nature of ettringite growth in this case was a function of the rapid supply of aluminate (rapidly dissolved from a relatively amorphous GGBS source) to combine with the other immediately available reactants thus permitting the rapid formation of ettringite in the controlled manner described by Little et al. (2010).

In terms of reactant timing, the oxidation state of sulphur is also of interest. Dependent upon the geological depositional environment, sulphur may be present as sulphate (e.g. gypsum Ca\(_2\)SO\(_4\)), or sulphide (e.g. pyrite FeS\(_2\)). While sulphates may release their SO\(_4^{2-}\) ions relatively rapidly into solution, the reduced sulphur in pyrite is unavailable for combination until it is oxidised. An oxygen rich environment, high pH, available water and Ca\(^{2+}\) is encouraged by the lime stabilisation process and facilitates rapid oxidation of pyrite to calcium sulphate (Casanova et al., 1997; Floyd et al., 2003). Hence, when assessing the sulphate swell potential of a soil, in addition to quantifying the immediately available sulphate, it is important to also determine the quantity of sulphate that may potentially oxidise from sulphide sources and combine these into a total potential sulphate (TPS) value (Longworth, 2004).

4.3.4 Summary of methods to control sulphate swell

To avoid sulphate related heave in lime stabilisation, the lowest risk approach involves avoidance of high sulphur clay soils and areas where groundwater may form a conduit for sulphate transport. A significant challenge is this regard is that sulphur bearing minerals are unlikely to be uniformly distributed through the host clay, and the initial identification of sulphur species may be missed at the site investigation stage. As a consequence, substantial advice has been published on suitable approaches to site investigation for identifying elevated sulphur content (e.g. Czerewko et al., 2003; Longworth, 2004; The Highways Agency, 2007) and UK formations that are known for their potential to contain elevated sulphur are well documented (Figure 6). A suggested safe maximum limit with the sulphur avoidance approach is 0.25% TPS, although this may be increased to 1% if soaked CBR tests do not indicate a swell
problem (The Highways Agency, 2007). However, it must be noted that the suitability of soaked CBRs to identify sulphate swell has been questioned (Highways Consultancy Group, 2008).

Where TPS levels above these low values are considered, any soil stabilisation must be considered an increased risk and the control strategies employed become a means of risk management. Regarding the previously noted strategies (see 4.3.1 to 4.3.3), some potentially complicating factors exist and this highlight areas where the direction of future research may assist the lime stabilisation industry. For strategies involving the deliberate growth of ettringite, the prolonged application of mellowing periods may result in significant clod strength before compaction takes place, and a high air voids percentage in the final compacted product. This conflicts with strategies seeking to improve durability by limiting access to water inflow; especially if this water contained further sulphate. The long term stability of ettringite is subject to environmental conditions promoting conversion to thaumasite. In this regard, the increased solubility of CO$_2$ in water of reducing temperature is considered as a possible trigger (Snedker, 1996; Collett et al., 2004). Therefore, a strategy pursuing the minimisation of air voids e.g., minimal mellowing and hydrated lime use, to avoid ettringite formation by denying reactant combination may seem preferable? However, future changes in environmental conditions, e.g. future addition of drainage trenches into the stabilised material, may trigger swell issues, for example by exposing / oxidising pyrite previously located deep within large clods (Floyd et al., 2003). The timely incorporation of industrial by products, e.g. GGBS, into the process may well alleviate some of these problems. For example, where air voids result from long mellowing periods, along with suppressing ettringite expansion, GGBS activation may promote the rapid growth of volumetrically stable reaction products to infill this inter-clod porosity.

It can be concluded that, in isolation, all of the discussed strategies would lessen the risk of deleterious mineral formation in the field. However, there would still be reasonably foreseeable field scenarios with the potential to instigate sulphate related attack and the most challenging scenario would be a highly plastic, high TPS clay. It may prove that a combination of the strategies into a specific working method may overcome this residual risk. Without further research work, this method is not clear. A further challenge is the absence of a satisfactory routine swell test for assessing the sulphate swell of site materials (Highways Consultancy Group, 2008). The soaked CBR test and accelerated swell test (BSI, 2004) are indicators of sample resilience to water and may indicate swell / strength reduction from water uptake by the clay minerals and, potentially ettringite formation from immediately available sulphate. However, as the potential triggers of sulphate swell - such as introduction of an oxidising/humid atmosphere (sulphide oxidation; Floyd et al., 2003) and a reduction in the soaking water temperature (e.g. for thaumasite growth; Snedker, 1996) are absent from these routine swell tests, there is presently no way for practitioners to adequately assess sulphate swell potential during pre-contract testing. Thus, an appropriate swell test remains a priority for industry (Snedker, 1996).
Figure 7. Distribution of UK strata that potentially contain sulphates and sulphides (DigMapGB-625, reproduced with the permission of the British Geological Survey © NERC. Contains Ordnance Survey data © Crown Copyright & database rights 2012).
5 SUMMARY

This paper has covered the key aspects relevant to the field practice of lime stabilisation and has highlighted that much of the subject is well understood and applied. Although, some routine practices e.g. mellowing periods, quicklime use and moisture conditioning close to the OMC may not always be suitable. If durability and/or long term performance are to be optimised, treatment of some materials (e.g. clay soils of medium – high plasticity) may require adaptations to these working practices. This is of particular relevance to high sulphur material, where minimisation of inter-clod air voids must be considered a primary defence against sulphate swell. Notwithstanding, there is still considerable research work which must be undertaken to identify the optimum working approaches for limiting sulphate swell risk. Fundamentally, future research must work towards addressing true industry need and the use of laboratory preparations which reflect site practice/conditions (Beetham et al., 2012).

5.1 Current understanding and required knowledge

The following bullet points summarise the key points discussed in this paper and also highlight where limited knowledge may be met with future research effort:

1. Other than initial access to very wet sites (which may use coarse lime nodules) current UK practice almost exclusively uses category 1 quicklime powder; the use of hydrated lime powder or lime slurries is rare.

2. The use of a small percentage of quicklime is well suited to some applications; specifically the rapid moisture conditioning of clay soils which will denudate to meet pulverisation specifications (i.e., The Highways Agency, 2009) with minimal mellowing period. This is ideal for a bulk fill compaction slightly wet of the OMC for MDD.

3. Irrespective of specifications that require a mandatory mellowing period of 24-72 hours (i.e., The Highways Agency, 2009), the inclusion of mellowing periods is not a straightforward issue. Mellowing periods of such long duration may permit clod strength to increase significantly before final compaction; leading to inter-clod air voids that persist throughout the compacted material.

4. Where quicklime (as opposed to hydrated lime powder/slurry) is used, the heat generated by slaking will further increase the potential for significant inter-clod voids and / or heterogeneous clod strength. This is a particular issue in lime treatment of clay soils of medium to high plasticity.

5. Minimising inter-clod air voids limits access to any soaking water; optimising durability and restricting volume change potential. The use of hydrated lime binders / slurries, minimal mellowing periods and a moisture condition significantly wet of OMC may minimise air voids, although the immediate strength may be less.
6. Longer mellowing periods (e.g. >12 hours) and quicklime may be suited to some applications. For example, where the mellowing period is followed by a second mix treatment of further binder e.g. Portland Cement. In such a scenario, the early lime-clay reactions improve pulverisation and reduce subsequent water demand from clods, then inter-clod cementation may both strengthen this region and infill some of the air voids. If the degree of inter-clod voiding is high (as may occur with prolonged mellowing (e.g., Sweeney et al., 1988) the degree of subsequent void infilling may be limited.

7. Current ‘state of the art’ demonstrates that lime migration may instigate diffuse cementation throughout larger clods of clay soil. The rate that strength gain is achieved with diffuse cementation is influenced by several factors and a high degree of soil saturation is of substantial significance.

8. The optimal combination of binders and different working methods for every different soil type cannot be known. This acknowledges the absence of a ‘one size fits all approach’ and the importance of selecting binder type, mellowing periods and moisture conditioning etc. based upon suitability to achieve the required engineering performance.

9. Our current specifications may be improved through allowance of a more flexible design/field approach, with facility for the controlled relaxation of some requirements. Specifically, mellowing periods and pulverisation should become appropriate to the material/binder type and whether the treatment intent is to promote a diffuse or inter-clod cementation. Pre-start, site specific mix designs that use a laboratory preparation method closely reflecting the intended field approach should play a role in controlling this (see Beetham et al., 2012 for an example of this preparation). A key focus of this approach would apply consideration of minimising air voids to maximise durability and mellowing durations should become an element of design and not specification.

10. The treatment of clay soils with a high TPS may result in sulphate swell. There are working methods which may reduce the risk of sulphate swell, however, our understanding as to which of these methods, or combination of these methods are durable over longer cures and environmental changes is limited. Future research should address this with an industry focussed approach, including development of a swell test which explores environmental changes.

11. Laboratory studies indicate that waste derived binders (e.g. waste paper sludge ash, red gypsum waste) have the potential to work as secondary binders within soil stabilisation. However, it is not clear how these may be incorporated into field applications. Required knowledge in this regard includes; how these binders influence diffuse or inter-clod cementation processes; durability implications and how specifications may be adapted to include their controlled use.
6 REFERENCES


APPENDIX B  NUCLEATION IN LIME STABILISED SOILS 
(PAPER 2)

Full Reference


Abstract

Nucleation centres, in the form of a small percentage of finely ground limestone powder, are often utilised to enhance the hydration of Portland cement. Cement mixing water has relatively low solubility and capacity to dissolve solutes from cement particles. Nucleation centres provide a surface of lower energy permitting hydration products to form in an expedited manner around these ‘seeds’. The mixing water is then able to further dissolve the cement particle and strength development of the hardening paste is accelerated. The laboratory study presented here investigates whether similar nucleation processes may prove beneficial to lime stabilised soils. One of the mechanisms driving the formation of cement product may be dissolution, from the edge sites of clay particles, of silica and alumina. These diffuse and combine with calcium and water forming cementitious minerals and associated strength gain. If this mechanism is present, then the solubility of the dissolving chemistries may cause a bottleneck and limit the rate of strength gain. It is possible that widespread nucleation centres may negate this bottleneck. This paper presents the initial findings of a practically focussed study into the potential of nucleation centres in lime stabilised soils.

Early results have identified that the use of clay clods up to 20mm in diameter, introduces a structure interlinking weak areas around clay clod peripheries and influencing the manner of sample failure. Pozzolanic reaction at the surface of the clods is considered to considerably influence this failure mechanism. CBR results (soaked and unsoaked) tested at 8 and 32 days suggest that there has been greater pozzolanic development due to the addition of finely ground limestone. However, due to the triaxial test results not supporting the CBR results and possible dry density influences, the results are equivocal at this stage. Further work to remedy this is underway.

Paper type – Conference
1 INTRODUCTION

Intermixing a small percentage of lime binder with poor quality host soils during earthwork construction has been employed as a ground improvement technique since the 1950’s (Petry & Little, 2002). Where clay minerals have a significant influence upon the engineering properties of a fill material, this addition of lime has a typically ameliorative effect. It is usual to discuss the effects that result from lime / clay reaction in terms of the time scale over which they occur. The short term effects relate to modification of the properties by drying (if quick lime is used) and cation exchange process. These benefits are often utilised as a construction expedient, therefore, a common and descriptive reference to these rapid changes is ‘lime improvement’. Where sufficient lime and clay reactivity is present, a secondary process involving time dependent, pozzolanic reactions may follow on. This growth of cementitious minerals further enhances the soils strength and durability (Sherwood, 1993) and may be given the amended distinction ‘lime stabilisation’.

The laboratory activities presented in this paper focus on gaining an enhanced understanding of the pozzolanic reactions which occur within lime stabilisation. This project is driven by the need to provide practitioners (the project sponsors) with information of practical benefit. Therefore, preparation techniques were applied that reflect the processes engaged on site. This, combined with the control and consistency of a laboratory setting, should provide a means of identifying mechanisms which may influence field behaviour.

1.1. Literature Information

1.1.1. Pozzolanic reaction

Lime stabilisation takes places through growth of a cementitious product. This is a reaction that primarily involves calcium, silica, alumina and water and in a high pH environment. The products are similar in nature to those derived from Portland cement hydration and provide the binders that keep the clay aggregations together (Greaves, 1996). The reaction products are usually described as calcium silicate hydrates or calcium aluminate hydrates (C-A-H). Other soil chemistries, such as sulphates, may alter the reaction products and cause potentially expansive minerals, such as ettringite and/or thaumasite to form (Hunter, 1988). For the purpose of this paper, such deleterious reactions are not specifically considered.

Whilst the pozzolanic reaction process is widely accepted as a concept, the detail surrounding a lime – clay cementitious reaction is poorly understood (Wilkinson et al., 2010). Boardman et al. (2001) noted a general lack of agreement amongst researchers, relating to the timescale and location of the reactions. This was attributed to the wide variety of clay soil compositions and complexity of clay mineral structures.
Two reaction processes have been discussed in the literature; a ‘rapid surface alteration’ and ‘long term dissolution and precipitation’. The rapid surface alteration process was proposed by Diamond & Kinter (1965) and considered that some initial pozzolanic reactions occur over a similar timescale to the lime improvement process and through the alteration of the surface of a clay particle. They suggested that calcium hydroxide is initially and rapidly adsorbed onto a clay particle’s surface. Cementation would then occur through Ca(OH)$_2$ reaction with Al(OH)$_X$ groups at the edge sites. The resultant C-A-H bridges and strengthens the edge / face contacts of the clays flocculated structure. This cementation at point of contact was considered by the authors to resist de-flocculation upon soaking, thereby accounting for the observed immediate improvement in swelling / plastic response to soaking.

Alternatively, dissolution and precipitation is a long term reaction, involving the release of aluminium and / or silica from clay particle edges under high pH conditions. New minerals are then precipitated at, or close to the dissolution sites (Diamond & Kinter, 1965; Sherwood, 1993; Boardman et al., 2001).

1.1.2. Nucleation in Portland Cement

In their state of the art review on “The Use of Limestone in Portland Cement”, Hawkins et al. (2003) summarise how finely ground limestone acts as nucleation centres within hydrating cement. They note how widespread distribution of the CaCO$_3$ centres accelerates the formation of hydration products, notably ettringite and portlandite. This expedited hydration removes solutes from the supersaturated mixing water and cement clinker particles may dissolve at a faster rate. Winter (2009) noted that this results in increased early (1-2 day) strengths and, due to a more uniform microstructure, late strengths (28 days) may also be enhanced.

1.1.3. Nucleation in Lime Stabilised Soils

In view of the above it is considered that finely ground limestone may have similar potential benefit as nucleation centres within the lime stabilisation process. Boardman et al. (2001) reported the presence of water soluble aluminium or silicon, as dissolved from the edges sites of kaolinite and bentonite. These ions were identified within 7 days of lime addition and their number had substantially increased by 175 days cure. With regard to new mineral growth, Boardman et al. (2001) considered that there was no sign of this occurring by 7 days, although substantial growth had occurred by 301 days. It may be considered, therefore, that dissolution of the necessary reactants occurs at a relatively early stage, yet mineral growth is not simultaneous.

If a contribution towards this delay is that the nucleation of the new minerals is slow due to high-energy requirements, then the provision of a low energy surface, as may be provided by well distributed, finely ground limestone, may encourage pozzolanic growth at an early stage. Such a mechanism may encourage widespread and accelerated cementitious mineral growth, with accompanying strength gain.
Reports of beneficial effects of nucleation surfaces in a lime-clay system have been supposed elsewhere within the soil stabilisation literature. This was made, however, with reference to the use of ground granulated blast furnace slag (GGBS) to mitigate the deleterious effects of ettringite formation in soils of high sulphate content. In such systems Wild et al. (1996) considered that GGBS provides an alternative nucleation surface that would compete with kaolinite to instigate ettringite growth. Ettringite grown from the GGBS particles was reported to have an absence of the property which caused it to secrete substantial H$_2$O and swell upon specimen soaking. Furthermore, in reference to pozzolanic reaction between lime and fly ash, Jalali et al. (1997) explained that an induction period observed in the strength gain of these mixes followed classic nucleation and diffusional growth theories. In consideration of reports that nucleation rates may influence strength gain and that reaction products may be nucleated at interfaces other than the clay particle surface, it was considered appropriate to trial the potential of finely ground limestone centres.

2 METHODOLOGY

2.1 Practical Considerations

Fundamental to this study was adopting preparation techniques which reflect the field practice of surface mixing lime stabilisation in the UK. This process uses mobile plant equipped with high torque rotational drums, arranged within a mixing chamber and appointed with multiple sharp teeth. The plant passes through a depth (nominally 300mm) of clay fill, and the action of the drums work to denudate the clay fill. Lime powder is added through an integral feed hopper. Mixing water may also be added via the mixing chamber. Upon completion of the mixing stages the binder should have been well distributed over the clod surface and, following any mellowing period requirements, the material is compacted with appropriate plant.

The clay clod sizes resulting from the above will be substantially reduced from their original size, however, there is often a practical limit to the degree of pulverisation that can be attained (Petry & Wohlgemuth, 1988), even with modern, high efficiency, machinery. Contractors are often required by specification to achieve a minimum degree of pulverisation e.g. the UK Highways Agency specification requires that 95% and 30% pass 28mm and 5mm sieves respectively (The Highways Agency, 2007). Hence, pulverisation beyond this minimum may not typically be pursued.

2.2 Material preparation

The aim of the trial was to compare and contrast the engineering properties of lime stabilised mixes made with and without nucleation centres. Additionally, a core focus of the laboratory trial was to engage in preparation techniques which are faithful to the site process. Specifically this involved utilising clod sizes achievable in the field, using a clay soil at ‘as dug’ moisture
contents and employing a binder addition / mixing process similar to field practice. Details of the clay soil are provided in Table 1. Both quick lime (Proviacal ST) and lime slurry (Proviacal SLS45) binders, supplied by Lhoist UK Ltd, were utilised. For the nucleation centres, a finely ground limestone was used, of Blaine surface area 480m$^2$/kg (Betocarb 80-BT supplied by Omya UK Ltd). The preparation procedure is detailed below.

Efforts were made to limit air drying effects which involved; use of a humidity room; clay storage in sealed sample bags and; at any interim periods of preparation, the clay clods were temporarily stored beneath a wet tented sheet, intended to provide a localised atmosphere of 95% R.H. The scale of test programme required that the test moulds were created in batches. This permitted all mould preparation to be completed within 2.5 hours of the final binder mixing stage (stage 5b in table 2). Each batch received identical preparation, undertaken in two stages; clay pulverisation / homogenisation and binder mixing.

| Description: Brown mottled grey soft to firm consistency low strength CLAY with rare fine to medium angular gravel of shale and rare rootlets. Sourced from a single trial pit at depths 1.5m to 3m below ground level within highly weathered Namurian age shale. Approx. Co-ordinates 40707 E 34979 N. |
| Mineralogy (as identified by qualitative XRD on air and oven dried specimen): Kaolinite, quartz, illite. Reflections between 14 Å and 10 Å also suggest an expansive clay mineral, possibly interlayered smectite–illite, however, this could not be fully determined from air / oven dried patterns alone. |
| Initial Consumption of Lime (from 3 tests). Average 3.53%. Range 3.4% -3.7% |

### Table 1 Details of the natural clay utilised in this study

#### 2.2.1 Clay pulverisation / homogenisation

The ‘as dug’ clay was pulverised by hand (small balls pulled away from the main clay mass). The resultant clods were passed through 20mm and then 14mm sieves. Each clod size group was thoroughly blended by hand. Each batch comprised 42.5kg of ‘as dug’ clay, which was composed of the same mass percentage of the two clod size groups i.e. <14mm = 13.7%; 14-20mm =86.3%, which were manually intermixed to ensure an even distribution.

#### 2.2.2 Mixing process

Table 2 summarises the process followed for each batch. The mixing times were considered sufficient to ensure that the binder/water was uniformly distributed over the surface of all clods. The total water added was determined through initial reference to the Moisture Condition Value (MCV; as described by British Standards Institution, 2002), with the intention of achieving a target MCV of 9.0. This MCV was selected because it should provide sufficient water to enhance ion transportation (Barker et al., 2007) and permit pozzolanic reaction products to form. This is in comparison to a drier material where both may become limited (Bell, 1988). Perry et al. (1996) note that MCV’s of 13.5 equate approximately with optimum moisture content (OMC), hence an MCV of 9.0 would certainly be wet of optimum, yet also workable...
on site. Once the mass of water required for this MCV was determined for the initial batch, this quantity was used for all subsequent batches. Batches were created for mixes with and without nucleation centres and for curing periods of 8, 32 and 189 days. Hence, there were a total of six batches treated with binder addition, along with a further batch of natural material (host soil) which was pulverised / homogenised, but not treated with lime.

2.3 Test programme

It was intended that cementitious mineral growth would be observed by increases in values recorded for CBR (soaked and unsoaked) and strengths determined from 38mm unconsolidated undrained triaxial tests. Whilst it is acknowledged that the CBR test is not a strength test in the strict sense, it does reflect the properties of stiffness, shear strength and deformation (Biczysko, 1996), hence an increase in CBR value is an indication in the beneficial promotion of one, or all of these attributes. For simplicity it will be referred to herein as a strength indicator. With a view to linking any observed increases in strength with changes in moisture content and/or plasticity, classification tests after each curing period were also undertaken.

For the natural (untreated) batch, sub samples were conditioned, either by air drying or with added water), to achieve four separate moisture contents ranging between 27.7% (approx. corresponding with OMC) to 38.7%. A similar range of tests were performed on the natural material, however, other than for targeted comparison with the stabilised mixes these results will not specifically be reported.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Description</th>
<th>Detail</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Binder 1 (quick lime) addition/ mixing</td>
<td>Quick lime addition to the average Initial consumption of lime value (3.53% by dry mass). Mixed for 10 minutes.</td>
</tr>
<tr>
<td>2</td>
<td>Mellowing</td>
<td>Clay clods thinned into an approx 50mm deep layer, covered with plastic and tamped. Material mellowed for 1 hour.</td>
</tr>
<tr>
<td>3</td>
<td>Disaggregation / Nucleation centres</td>
<td>Any lightly adhered clods separated manually. For mixes containing nucleation centres, 3.5% by dry weight was evenly spread over the surface.</td>
</tr>
<tr>
<td>4a</td>
<td>Binder 2A (SLS45)</td>
<td>Lime slurry addition in quantities to equal the same molar mass of CaO added in stage 2 (i.e. equivalent of 3.53% quicklime) Mixed for 6 minutes.</td>
</tr>
<tr>
<td>4b</td>
<td>Binder 2B (SLS45)</td>
<td>Further lime slurry added to bring total lime slurry addition to equate to an equivalent of 5% quicklime. Mixed for 4 minutes.</td>
</tr>
<tr>
<td>5a</td>
<td>Water A</td>
<td>50% of the mixing water added (523ml). Mix for 6.5 minutes.</td>
</tr>
<tr>
<td>5b</td>
<td>Water B</td>
<td>Remaining 50% of mixing water (523ml) added. Mix for 6.5 minutes.</td>
</tr>
</tbody>
</table>

Table 2 The preparation stages engaged to replicate the site process

2.4 Curing and Testing

All curing (in air or in water) was undertaken at a temperature of 20°C +/-2. All exposed sample surfaces were wrapped with several layers of cling film and the edges were sealed by parcel
tape immediately after trimming to test dimensions. This was to prevent evaporation during curing. For CBR mould soaking, the process described by British Standards Institution (2004) was followed with the exception that the sample’s top surface was left exposed to the water. The top perforated plate / surcharge rings were omitted as this comprises a more aggressive durability test. Soaking durations were 4 days for the 8 day cure samples and 28 days soaking for all others. Apart from stated deviations, testing was undertaken in accordance with the appropriate current British Standards. For the triaxial tests on stabilised samples, in order to reflect the near surface environment of their typical application, cell pressures of 25, 50 and 75 kN/m$^2$ were used.

The following reference system is used: [Days cured]_[Test name (cell pressure)]_[If soaked]_[Binder Type] e.g. 7_CBR_S_Nuc or 32_TRI(50)_L.

## 3 RESULTS

At time of submission, results from the 8 day and 32 day curing periods were available. The 189 day cure results will be available by and presented at the IS-GI 2012 conference.

### 3.1 Classification tests

Table 3 contains the classification test results. This demonstrates that the mixing process increased the moisture content from ‘as dug’, however, it is apparent that the results of nucleation centre batches are, on average, 1.8% lower than the lime only mixes. This correlates with the reduction that would be expected from the extra 3.5% of solid mass added with the nucleation centres. In all cases the moisture content reduces following air curing and by 32 days this reduction was approx. 0.8%. Soaking appears to reinstate the moisture content to a similar, or slightly higher, level than the mixing value.

<table>
<thead>
<tr>
<th>Batch</th>
<th>Binder Mixing</th>
<th>Air Cure</th>
<th>Soak Cure</th>
<th>Difference between mix and air cure</th>
<th>W_P</th>
<th>W_L</th>
<th>I_P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural</td>
<td>Average natural moisture content = 35.4%</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>8_L</td>
<td>38.8</td>
<td>38.6</td>
<td>38.9</td>
<td>-0.2</td>
<td>48</td>
<td>69</td>
<td>38</td>
</tr>
<tr>
<td>32_L</td>
<td>38.8</td>
<td>38.0</td>
<td>39.4</td>
<td>-0.8</td>
<td>51</td>
<td>87</td>
<td>35</td>
</tr>
<tr>
<td>8_Nuc</td>
<td>37.3</td>
<td>36.7</td>
<td>37.4</td>
<td>-0.6</td>
<td>47</td>
<td>85</td>
<td>38</td>
</tr>
<tr>
<td>32_Nuc</td>
<td>36.7</td>
<td>35.9</td>
<td>37.0</td>
<td>-0.8</td>
<td>46</td>
<td>80</td>
<td>34</td>
</tr>
</tbody>
</table>

Table 3  Average moisture content and Atterberg limits for the binder treated batches. Atterberg’s for natural material are shown for comparison. Moisture contents are the average of at least 4 tests (binder mixing) and up to 9 tests (cured batches). Atterberg’s are the average of 2 (cured batches) or 3 (natural material) tests.

Following 8 days curing, liquid and plastic limits for both mixes had increased by 15-16%, which resulted in an effectively unchanged plasticity index (I_P) of 37-38%. The 32_L results indicated slight further increases in liquid limit (W_L) and plastic limit (W_P) combining to reduce
I_P to 35%. The 32_Nuc results had a similarly reduced I_P of 34%, however, this was manifest through a small reduction in W_P with a notable reduction in W_L to 80%.

3.2 **Strength Test Results**

3.2.1 **CBR**

Table 4 illustrates the CBR results for all tested specimens. As might be expected, average CBR values for all specimens increase with prolonged curing. For both curing periods and conditions, the samples with the nucleation centres have higher CBR values than their lime only counterparts. The difference is most notable when comparing the 32 day soaked specimens, with the nucleation samples average CBR of 14.7% being 28% greater than the lime only average of 11.5%.

<table>
<thead>
<tr>
<th></th>
<th>Air Cure CBR (%)</th>
<th>% Increase from 8-32d (ave)</th>
<th>Soaked CBR (%)</th>
<th>% Increase from 8-32d (ave)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Top</td>
<td>Bottom</td>
<td>Ave</td>
<td>Top</td>
</tr>
<tr>
<td>8_CBR_L</td>
<td>9.7</td>
<td>12.0</td>
<td>10.9</td>
<td>9.4</td>
</tr>
<tr>
<td>32_CBR_L</td>
<td>14.2</td>
<td>15.1</td>
<td>14.7</td>
<td>10.0</td>
</tr>
<tr>
<td>8_CBR_N</td>
<td>11.7</td>
<td>13.1</td>
<td>12.4</td>
<td>10.1</td>
</tr>
<tr>
<td>32_CBR_N</td>
<td>15.0</td>
<td>18.3</td>
<td>16.6</td>
<td>12.8</td>
</tr>
</tbody>
</table>

Table 4 Average CBR results for all tested specimen. All results are the average of 3 separate tests.

3.2.2 **38mm unconsolidated undrained triaxial tests**

Table 5 summarises the peak shear stress recorded on failure of the triaxial specimens and Figure 1 presents the stress / strain plots. For all samples the shear stress at failure increases with curing period and confining stress. For the 32 day cure samples, the strain at peak strength was notably reduced from those at 8 days. At 8 days cure it is clear that the nucleation samples are of superior strength. By 32 days, however, there is less discernable difference, especially at 75kN/m$^2$ confining stress, where shear strengths are almost identical at 154 and 156kN/m$^2$. This is considered further in Section 4.

<table>
<thead>
<tr>
<th>Confining pressure (kN/m$^2$)</th>
<th>Shear Stress at failure</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>25</td>
</tr>
<tr>
<td>8_L (kN/m$^2$)</td>
<td>85</td>
</tr>
<tr>
<td>32_L (kN/m$^2$)</td>
<td>122</td>
</tr>
<tr>
<td><strong>8-32 d increase (%)</strong></td>
<td></td>
</tr>
<tr>
<td>8_Nuc (kN/m$^2$)</td>
<td>112</td>
</tr>
<tr>
<td>32_Nuc (kN/m$^2$)</td>
<td>130</td>
</tr>
<tr>
<td><strong>8-32 d increase (%)</strong></td>
<td>16</td>
</tr>
</tbody>
</table>

Table 5 Triaxial test - shear stress at failure
3.3 Sample Structure

3.3.1 Triaxial tests
Observation of failure planes that developed during the triaxial tests on the binder treated samples indicated that the structure of the sample may have influenced the mode of failure. Figures 2a-d illustrate examples of the types of failure planes identified in the samples. Failure surfaces were typically rough, possessing either undulating, wavy or a stepped / jagged profile. Occasionally flat smooth areas were present, sometimes with striations (slickensides). Figure 2.a is an atypical example where slickensides have developed over much of the failure plane surface. The failure plane of some samples change considerably i.e. from a shallow to steep angle from the horizontal. This is either through abrupt angle changes (figure 2.b) or with a more gradual / curving profile (figure 2.d). Occasionally failure planes were relatively straight (figure 2.c) although often minor stepping / jagged areas were still present. Other specimens displayed even more complex planes. Figure 2.a, illustrates a failure surface which appears relatively straight/diagonal on one side, yet with distance into the sample, rotates both out of plane and steepens to daylight in an irregular manner on the opposing side.

3.3.2 CBR Tests
Following completion of the CBR tests, the samples were broken up for classification testing. During this process for the 32 day cured samples, the presence of numerous isolated nodules
Figure 2 a-d Triaxial sample failure planes evidencing a macrostructure within the sample. All photos were oven dried at 105°C prior to the picture being taken.

Figure 3 a (left) ‘Stiff’ nodules recovered from 32_CBR_L. 2b (right) A nodule split in half.

surrounded by a matrix of softer material were identified. It was possible to scrape away the softer matrix to reveal the nodule’s profile (figure 3.a). The larger nodules could be split in half with strong finger pressure (figure 3.b), which revealed they comprised intact clay material and not agglomerates of smaller clods. The nodules were typically stiff and friable which caused them to appear of lower moisture content than the relatively plastic matrix. The nodules were typically elongate and sub rounded with the long axis ranging in size from 10mm to 30mm. These features were present within lime and nucleation mixes and in the air cured and soaked
CBR specimens. Although no attempt was made to measure this, it could be estimated that the nodules comprised approximately 30% of the sample volume. It was also considered that the nodule representation appeared greater in the nucleation samples. However, in the absence of measurement, this is subjective. No nodules were observed in the 8 day cured samples, although, as they were not specifically sought at that stage, this does not evidence their complete absence.

4 DISCUSSION

If the finely ground limestone centres were performing the function of nucleation centres, it may be anticipated that there would be a difference in strength values at an early stage, i.e. the 8 and 32 day curing periods selected. Indeed, the 8 day results reveal that all strength indicator tests for the nucleation centre mixes are greater than their lime only equivalents. This might be considered evidence of early nucleation. However, it may alternatively be considered that the strength difference is a residual function of the nucleation samples having a lower moisture content and therefore, higher dry density at preparation.

The 32-day results were less conclusive. The percentage increase in CBR results between the 8-32 day curing periods for both lime only and nucleation samples were near identical at 34% and 35%. Therefore, the nucleation specimens maintained the notably superior values identified at 8 days. The triaxial results, however, reveal that the lime only samples experienced a greater rate of strength gain (ca. 45%), than the lesser increase (16-27%) observed for the nucleation samples. Accordingly, whilst the nucleation samples are typically of greater shear strength than the lime only, this is less marked at the lower confining stress and is not evident at 75kN/m² cell pressure. Therefore, whilst the CBR results provide an indication that the nucleation mixes are stronger and certainly more durable to soaking, the triaxial tests do not provide complete agreement.

The consistency tests reveal some small changes with time. Most notably $W_L$ and $W_P$ results at 32 days are 5% and 7% lower (respectively) for the nucleation specimens. This difference may indicate different reaction rates. Literature reports on the change of Atterberg limits in response to lime treatment are variable and tend to focus on the modifications process. However, of the limited comments on Atterberg response over time, Sherwood (1993) suggests $W_P$ and possibly $W_L$ will increase as pozzolanic reactions develop. As the $W_L$ for the nucleation specimens appear to be reducing with curing time, strength increase and (presumed) pozzolanic reaction, this is an interesting contradiction.

It is suspected that the reason for a lack of clarity surrounding the results is the influence of the observed structure. For the natural clay samples (particularly the driest), it was suspected that the clod boundaries comprised a weakness which influenced failure. For the stabilised samples, clod boundaries may also comprise a weakness. However, the identification of hard nodules
within the CBR samples suggests the shear strength would also be zonal, where an apparently weaker matrix separates the stiff nodules. With this structure, the failure planes would essentially link points of pre-existing weakness, stepping around strong locations, resulting in the irregular profiles described. Picarelli et al. (1988) noted that a similar mechanism occurred during undrained triaxial tests of an over-consolidated clay shale. Pre-existing planes within samples of this clay shale were considered to act as weaknesses, along which shear failure mobilised, but only following an initial period of high stiffness at small strains. At these small strains, it was considered that stress was relatively uniformly distributed through the entire sample. With increasing stress, the strain became localised at the weak planes and a dramatic reduction in stiffness occurred. Picarelli et al. (1998) considered this rupture mechanism to be analogous to the failure of a jointed rock, where mass strength is dominated by the frictional resistance of the discontinuity and not by strength of the solid rock. This rock mechanics analogy may be completed by consideration of Hoek’s (2007) explanation that peak strength equates to the sum of cementing forces holding rock surfaces together plus frictional resistance. Hence, the stabilised soils would have a minimum ‘cohesion’ plus an increasing frictional contribution to shear strength with confining pressure. Such an increase with confining pressure is reflected by Figure 4.

It has been suggested that the stiff nodules have influenced the profile of the failure planes. Therefore, it is worthwhile considering how they developed. Petry & Wohlgemuth (1988) proposed that pozzolanic reactions develop a coating on the surface of clay clods, which, in their study was considered to provide substantial durability against wetting/drying effects. It is possible that the stiff nodules noted in the present study have developed where pozzolanic reactions at the clod surface have essentially segregated the interior of the clod from the rest of the sample. This closed system appears to have been subject to different influences, with the internal region developing observably different engineering properties (stiff / friable) when compared with the matrix. The stiff nature of the nodule interior may be due to a lower moisture content and/or soil suction effects. Such a situation may be generated by the consumption of H$_2$O in the formation of the cementitious product at the periphery. Pozzolanic reaction within the nodule interior is another possibility. With a coating mechanism, it would be anticipated that where pozzolanic reaction has occurred at an accelerated rate, resilience to soaking would result. This may account for the superior soaked CBR values of the nucleation samples, thus potentially evidencing the nucleation process.

It is suspected that further and widespread pozzolanic reaction at clod peripheries would cause the stiff nodules to increase in representation. By inspection this would lead to a reduction in the number of weak zones in the sample and sample strength would increase accordingly. If cementitious reaction is also able to bond adjoining clod surfaces together, then this may equate to the point where the underlying structure is overcome and the intact strength of the stiff clods would dominate. In such a scenario it would be anticipated that an inclined failure plane would shear triaxial samples at a relatively consistent angle, as might be anticipated for a ‘normal’
brittle failure. It will be interesting to see if this stage, or notable progression towards it, is evidenced in the 6 month results.

In view of the original aim, there is some suggestion in the CBR results, particularly the soaked CBRs, of early nucleation providing increased strength in the samples treated with finely ground limestone. Notwithstanding, the described inter-clod structure has provided weak planes, which appear to have influenced the results, in particular those of the triaxial tests. There is also the possibility that different moisture contents on preparation may account for some of the superior performance in nucleation samples. It may prove that with further curing time, i.e. upon testing of the 6 month cured samples, the strength difference and role may be clearer. It is necessary to use additional methods of enquiry in order to determine whether finely ground limestone do act as nucleation centres. The following additional works are currently underway:

1. Scanning Electron Microscopy analysis; to provide visual evidence (or otherwise) at a microscopic scale, of preferential nucleation around the limestone particle;
2. Further triaxial test specimens prepared at comparable moisture contents and cured for 8, 32 and 189 days. Intended to remove the moisture content anomaly and provide further information on the development of failure plane surfaces.
3. X-Ray Diffraction analysis of 32 and 189 day cures. To identify growth of new mineral phases.

5 CONCLUSION

This paper has investigated the potential role that finely ground limestone, intermixed within a lime stabilised soil, may have as nucleation centres. An approach to producing test specimens in a manner which closely reflects UK site practice has also been described. The following conclusions are taken:

- The preparation method employed resulted in formation of a structure which appeared to influence the failure of all specimens. This structure was considered to form through the interlinking of weak areas around clay clod peripheries and its influence was observed through unusual failure planes in the triaxial specimens;
- Following 32 days curing, the development of stiff, friable nodules within a matrix of softer material was noted within all CBR mould specimens. The nodules were considered to have developed where pozzolanic reaction had occurred at the periphery of a clay clod. This coating appears to have led to enhanced engineering properties developing within the clod interior;
- Superior CBR values (most notably the soaked values) in the limestone treated specimens, may reflect the role that further and more widespread nucleation plays in enhancing the growth and distribution of the (above described) pozzolanic coating;
- At this stage it is not possible to conclude that finely ground limestone do provide a beneficial role as nucleation centres under the conditions investigated. This is due to the
triaxial test results not supporting the CBR results and possible dry density differences between the samples. Further works to remedy these conflicts is underway.

The authors would like to thank the project sponsors (Engineering and Physical Sciences Research Council, Opus International Consultants and the Independent Stabilisation Company) and the companies supplying materials (Lhoist UK, Omya UK and Lafarge Cement UK).

6 REFERENCES


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APPENDIX C LIME DIFFUSION AND IMPLICATIONS FOR LIME STABILIZATION PRACTICE (PAPER 3)

Full Reference

Abstract
This paper summarizes efforts to improve understanding of how the inclusion of clay clods up to 20mm in diameter influences the strength of a lime stabilized clay soil of moderately high plasticity. Findings demonstrate that Ca$^{2+}$ ions and hydroxyl groups do migrate deep into these larger clods, forming a calcium-silicate-hydrate gel which coats the surface of clay mineral aggregations within these clods, to increase cured strength over 8, 32 and 194 days. Moisture conditioning to wet of the optimum moisture content for compaction was considered to enhance the ion diffusion process.

While pozzolanic reaction increases the strength of the clods, air voids located between clay clods act as weaknesses that play a significant role in reducing specimen strength throughout all curing periods. This was particularly pronounced where sample curing involved a soaking phase as these air voids transmit water to weaken the surface of the clay clods. The application of a mellowing period was considered to increase this negative effect.

Paper type – Conference
1 INTRODUCTION

Stabilization, or the improvement in engineering properties of soft clay soils can be achieved through inter-mixing with lime. Application of lime stabilization during earthworks construction involves the use of large mobile plant to firstly rotavate the clay soil to smaller clods and enable the distribution of lime over these clod surfaces. This may then be followed with stages of mellowing (a delay of specified duration before the next stage), further binder/water addition and mixing, before final compaction of the treated material.

The size of the clay clods, or degree of pulverization, generated by this process is considered important for the effectiveness of lime-stabilization. Therefore, construction specifications often place an upper limit on the maximum permissible clod sizes. For example, the United Kingdom Highways Agency specification, which forms the basis of most soil stabilization works undertaken in the UK, requires 95% of the treated material to pass a 28mm sieve and 30% to pass a 5mm sieve [1].

In stark contrast to the practical realities of working with relatively large clods, a high proportion of the lime stabilization literature is based upon laboratory experiments on very finely pulverized clays [2]. The few publications that have investigated the influence of different clod sizes (such as Petry and Wohlgemuth [3]; Bozbey and Garaisayev [2] report that the finest clod sizes (<5mm) are preferable for maximum strength and durability. Petry and Wohlgemuth [3] suggested that the centers of larger clods did not react at all and that pozzolanic reaction was restricted to locations close to the clod periphery. The conclusion of Bozbey and Garaisayev [2] was that field applications should seek to minimize clod size and avoid the inclusion of large clods; but this may be unrealistic.

Ion diffusion is capable of driving pozzolanic reactions into the core region of clods up to 20mm in size [4, 5]. The efficiency of this diffusion process is paramount to facilitate the migration of calcium ions and/or hydroxyl groups (OH−) over distances of tens of millimeters before chemical change may occur at these clod centers. This process appears to be most efficient at the liquid limit [6]. However, in terms of a practical application, operating plant at this soil state is prohibitively difficult and expensive.

In summary, it can be observed that:

1. Practical applications of in situ lime stabilization involve clay clod sizes many times larger than the fine gradations investigated and advocated by the academic literature;
2. Ion diffusion and pozzolanic reactions are most efficient at high moisture contents, but in situ mixing at the liquid limit is not economically feasible;
3. There remains a gap in knowledge relating to the development of pozzolanic reactions deep into clay clods, particularly those prepared at conditions wet of OMC.
1.1 Background

This paper reports a laboratory investigation conducted upon lime treated clay clods (up to 20mm in size and conditioned approximately 1.15 times wet of OMC using methodology in line with industry practice for in-situ lime stabilization) compacted and cured for 8, 32 and 194 days.

Some results from early cures were reported in a previous publication i.e. Beetham et al [7] and the key findings from this are summarized: Strength tests on the 8 and 32 day cures revealed that a general increase in cured sample strength did not develop in a homogenous manner. At 32 days the strength of some clods had increased significantly, whereas the strength in other clods had increased to a lesser degree. The clods which had increased in strength appeared of consistent strength throughout, i.e. an absence of soft center / hard periphery. There also appeared to be a fine air gap separating neighboring clods (an inter-clod porosity). Failure planes were noted to propagate through the weakest points ‘stepping’ around the stronger clods. It was suspected that this heterogeneous strength development indicated something about how/where pozzolanic reactions had progressed, although the underlying detail was not clear. The present paper adds further strength tests for all cure periods along with a detailed chemical and physical investigation into the 194 day cured specimens.

Therefore, the present paper substantially updates findings from the Beetham et al paper [7] and delivers an improved understanding of how lime migration progresses pozzolanic reactions throughout clods of moderately high plasticity clay.

2 METHODOLOGY

2.1 The Test Clay

The clay used for this study was sourced in an ‘as dug’ state from a single trial pit. The clay mineralogy was identified by X-ray diffraction (XRD) and comprises kaolinite, quartz, illite and an expansive clay mineral (suspected to be smectite-illite interlayers). The plasticity index was 38% (plastic limit of 31%; liquid limit of 69%) and the in situ moisture content was 35.4%. The initial consumption of lime (ICL) value was 3.5% by dry mass of soil [7]. Total chemistry determinations by X-ray refraction revealed a very low calcium content (CaO = 0.25% by mass).

2.2 Sample Preparation

The mix preparation process was developed in great detail to represent methods employed on site, and these are described more fully in Beetham et al. [7]. For the purpose of this paper a brief summary is provided below.

A key aspect in the replication of the site approach was to process the clay ‘as dug’, i.e. at the in situ moisture content. Multiple batches of materials were prepared using a common method. This involved pulverization of the clay in a consistent manner, with each batch comprising 86% of clay clods in the size range of 14-20mm, and 14% of clay clods smaller than 14mm in size.
Lime was added at two separate mixing stages; at the first stage quicklime was added in a quantity equal to the ICL value of 3.5%. The mix was then sealed and allowed to mellow for 1 hour. At the second mixing stage, hydrated lime slurry was added (equivalent in mass to a further 5% of quick lime) and a fixed amount of water was added to the mix, sufficient to achieve the desired moisture condition value (1.15 times OMC).

Immediately following mixing, the treated material was compacted, using standard 2.5kg proctor effort, into California Bearing Ratio (CBR) or proctor moulds. Specimens of 38mm diameter (for triaxial testing) were then extruded from the proctor moulds. Following trimming to test dimensions, samples were sealed and allowed to cure in a temperature and humidity controlled environment at 20°C. Most samples were cured in air (unsoaked), although some of the CBR specimens were fully immersed in water (soaked) for the latter portion of the cure period (Table 1).

2.3 Strength and Atterberg Limit Tests

Upon completion of curing, 38mm unconsolidated undrained triaxial tests and CBR tests were executed in accordance with the relevant British Standard [8, 9]. The triaxial tests were carried out at low confining stresses (to replicate near surface conditions), ranging from 10 to 75 kN/m² and at a strain rate of 1.2mm/minute. Atterberg tests in accordance with the British Standard [10], were undertaken on representative material recovered from the tested CBR’s.

2.4 Detailed Investigation at 194 days

Following completion of the 194 day CBR tests, the complete specimen was extruded from the mould for careful examination. As with the 32 day cures [7], it was clear that the CBR specimens were composed of individual clods closely separated by a narrow, inter-clod pore space. The physical arrangement of the clods within the mould was recorded before careful separation (using finger pressure) of clods from their neighbors. The recovered clods were then passed through 20mm, 14mm and 10mm sieves so that larger / smaller clods could be analyzed separately. The total weight and moisture content of each clod sizing were then determined for both soaked and unsoaked cures.

To investigate differences between the outer and inner areas of individual clods, a soil shave was used to remove the outer 2-3mm from clods in the 14-20mm clod size fraction. Following careful removal of the outer clod material, avoiding contamination, the remnants of the intact clods were then used to provide samples from the ‘inner’ clod. To attain sufficient sample it was necessary to repeat this process on 8 separate clods.

Subsequent chemical analyses were undertaken on representative material, either from the whole clods, or the outer/inner clod fractions and using the below methods:

2.4.1 pH and XRD

Batches of material were oven dried at 40°C and ground to a fine powder with pestle and mortar before testing for pH (in accordance with [10]) and analysis by X-ray Diffraction (XRD). XRD
scans were run on a Bruker D8 powder diffractometer with a copper source, which analysed 20 angles between 5° and 65° for a run time of 2 hours.

2.4.2 ICP-AES

Determinations of total calcium content were made by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES). Prior to analysis, samples of oven dried (105°C) and finely ground material, were microwave digested within aqua-regia. Solutions were then filtered and diluted in pure water to a precise volume before analysis using a Shimadzu ICPE 9000.

2.4.3 SEM-EDX

Micro-chemical determinations were undertaken using a Cambridge S360 scanning electron microscopy (SEM), with Oxford Instruments x-ray detector (EDX), at an accelerating voltage of 20kV. Samples which exposed a complete cross section through the middle of large (ca. 20mm) clods were prepared, thereby allowing direct comparison of regions from the very periphery with those from the center of the same clod. A highly polished / flat surface was achieved over this cross section by following a procedure of resin impregnation / polishing (as described in Appendix B of Winter, 2012 [11]), thus facilitating accurate quantitative micro-analysis [11].

3 STRENGTH AND ATTERBERG RESULTS

3.1 Strength Test Results

The CBR test values increased with progression of the curing period. The CBR values for samples cured in air attained an average of 20.8% at 194 days curing. The effect of soaking on the CBR values was significant, with the 32 and 194 day cures only reaching approximately 80% of the unsoaked values (Table 1).

The triaxial test results show significant strength gains with increasing curing time (Figure 1). For example, 194-day samples, tested at a confining stress of 25kN/m², reached a peak shear stress of 617 kN/m². This is more than three times the value attained at 8 days (Figure 1).

The curing process gradually changes the failure behavior of the materials. The earlier cures (8 and 32-days) can be characterized by a degree of strain hardening. However, the 194-days cured samples display a pronounced brittle failure. The rapid decline in post-peak shear stress

<table>
<thead>
<tr>
<th></th>
<th>8 Day</th>
<th>32 Day</th>
<th>194 Day</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unsoaked CBR (%)</td>
<td>10.9</td>
<td>16.6</td>
<td>20.8</td>
</tr>
<tr>
<td>Soaked CBR (%) (days soaked in parentheses)</td>
<td>10.3 (4d)</td>
<td>11.5 (28d)</td>
<td>14.7 (28d)</td>
</tr>
<tr>
<td>Plastic Limit (%)</td>
<td>47</td>
<td>51</td>
<td>51</td>
</tr>
<tr>
<td>Liquid Limit (%)</td>
<td>85</td>
<td>86</td>
<td>82</td>
</tr>
<tr>
<td>Plasticity Index (%)</td>
<td>38</td>
<td>35</td>
<td>31</td>
</tr>
</tbody>
</table>

Table 1 CBR Values (averages of three samples) and Atterberg Limits (average of two tests) For All Cure Periods
suggests that a sudden release of particle interlocking or severance of cemented bonds occurs upon failure. In general, it can be observed that peak shear stress increases with confining stress. However, some test results deviate from this trend. Furthermore, stress-strain plots illustrate a degree of inter-sample variability. It is possible that these anomalies were due to inter-clod porosity causing markedly lower peak shear stresses being mobilized [7]. These observations point towards the combined importance of pozzolanic reactions and sample structure on the mobilization of shearing resistance for later discussion.

3.2 Atterberg Limits

Atterberg limits displayed an initially rapid increase in the plastic and liquid limits by 8 days, but further curing of the samples resulted in little change in the plastic limit. The liquid limit saw a slight reduction from the 8 to 194-day cure periods. As a consequence, the plasticity index reduced from 38% for clay in its natural state; to 30% for the clay at 194-days curing time (Table 1).
4 INVESTIGATION OF 194 DAY CURED CLODS

4.1 Observations

Inspection of the ‘post-test’ material extruded from the CBR revealed several important aspects relating to the overall sample structure.

Breaking up of the samples was possible along weaknesses due to the inter-clod porosity. All clods recovered from the sample were of substantial strength and it was not possible to ‘fracture’ individual clods with finger pressure. Cross sections through the clods could be formed with a soil grater and did not reveal any obvious differences in strength between inner/outer regions. Clods were typically angular and arranged in close proximity to adjacent clods. This general configuration was likely achieved during compaction, where initially soft clay clods would remold around one another. As the clods had developed strength with curing, the overall effect at 194 days was that of strong clods ‘inter-locked’ around their neighbors. Occasionally, smaller clods (i.e. 1-5mm diameter), located between the larger clods were observed to lessen the degree of interlocking. These small clods had not been remolded to the same extent as the larger clods and the inter-clod porosity was notably increased at these locations (See Figure 5). Thus, the sample parted easily around these locations of higher porosity, separating into smaller blocks of interlocked clods. The clods within these blocks could usually be separated from their neighbors with finger pressure. However, while only light finger pressure was required to deconstruct the soaked samples into single clods, the unsoaked samples required stronger pressure and some could not be manually parted at all (This artificially increased the >20mm fraction; Figure 2). This indicates that significant soil suctions were acting between adjacent clods in the unsoaked cures and that these were removed by soaking.

4.2 Moisture / Calcium Distribution Over Clod Sizes

As expected, the lime treatment, 1-hour mellowing period and subsequent mixing process was effective in increasing pulverization and the total percentage of clods >14mm was considerably less than the original clay pulverization (Figure 2). Furthermore, a significant class of fines (<10mm) had developed. Hence, it is likely that these fines were created by the removal of material from the periphery of originally larger clods.

The ICP analysis reveals that the average calcium content in the <10mm fraction was significantly greater than any other size gradation (Figure 3). Thereafter, the results may be considered surprising as the largest clods possess the second highest concentration of calcium. Results for soaked and unsoaked cures are comparable which suggests the soaking process did not leach significant quantities of calcium from the specimens.

The soaked samples have greater moisture contents than the unsoaked cures (figure 3). Considering the soaked samples were completely immersed for 28 days, the moisture increases are modest, especially for the larger clod intervals. An increasing difference in moisture content (1.5% rising to 4.5%) with decreasing clod size may reflect the relatively larger contributions
of ‘surface area versus volume affected’ for the smaller clod. Additionally, a substantially higher unsoaked moisture content for the <10mm fraction may indicate that these fines have different properties as they are predominantly produced during sample mixing and were directly exposed to higher concentrations of lime and mixing water (Figure 3).

Figure 2. Size gradations of the clods recovered from the soaked / unsoaked 194-day CBR’s. All material recovered from each CBR was sieved in this manner, thus each plot is the average of three separate gradings.

Figure 3  Moisture and calcium distribution of each clod grade recovered from the soaked / unsoaked 194-day CBR’s. Each plot point is the average of at least three separate tests.
4.3 Lime Migration / Chemical Change Throughout Clods

4.3.1 Calcium and pH analysis
Average values from four tests revealed that the outer region of the 14-20mm clod gradations were characterized by a pH of 12.44 +/-0.1; a value consistent with the maximum attainable from the added lime. The inner regions were characterized by a pH of 11.85 +/-0.05, i.e. not quite as high as the peripheral zones but still substantially higher than the pH of the source clay (pH 5.39-6.0).

A similar situation was observed with calcium determinations, whereby both regions experienced a substantial increase, although the calcium concentration in the outer clod was approximately 80% higher than the inner (Figure 3).

These pH and calcium gradients, confirm that a migration of Ca$^{2+}$ ions and hydroxyl groups (OH$^-$) had taken place from the periphery into the core.

4.3.2 XRD
To reveal crystallographic modifications, comparison of the data from the clods with data from the source clay was undertaken. The samples from outer clod areas showed strong new peaks at 2θ spacings of 34.09, 18.09 and 47.12. These are consistent with Portlandite (Ca[OH]$_2$). These new spacings were absent in patterns from the inner core. Otherwise, no peaks additional to those also noted in scans on the source clay were identifiable.

4.3.3 SEM-EDX
The objective of this analysis was to identify the micro-scale distribution of calcium. The very low calcium content in the source clay suggests that consistent and substantial determinations of calcium must be due to the added lime.

The most striking features identified by the SEM analysis on 194 day cures were accumulations of new formed minerals noted in a thin veneer within 1mm from the clod edge. These micro-concretions had a distinctive pseudo-spherical shape, ranged in diameter from 1 – 100µm and were predominantly composed of Calcium Silicate Hydrate (See Figure 4 and Table 2), although inclusions of Portlandite (up to 10µm) were noted. The Ca/Si atomic ratio of the C-S-H was typically 1.1-1.3 (although occasional small inclusions of Portlandite within the analysed volume caused local increases). Minor quantities of Aluminum and Iron were also identified within C-S-H micro-concretions.

SEM analysis undertaken at 32 days also identified micro-concretions close to the clod edge. However, they were less in number and size (maximum 50 µm diameter). Again, the composition were typically C-S-H, however, rare examples were composed only of Portlandite.

While interesting features, the micro-concretions were relatively minor in occurrence; even at locations close to the clod edge. The typical field of view was relatively consistent throughout all regions of the clods (i.e. both inner and outer clod locations were very similar) and comprised a matrix of fine alumino-silicate materials with larger clasts (shown in figure 4). The clasts
Figure 4  Example SEM BSE image from near clod surface region of 194 day cure. The scale bar indicates the dimensions of three pseudo spherical C-S-H micro-concretions (surrounded by annular porespace - from shrinkage due to specimen drying) separated by matrix. The locations of analysed spectra within and around the C-S-H micro-concretions are indicated by numbered arrows and the corresponding elemental analysis in Table 2 distinguishes matrix regions from the C-S-H micro concretions.

<table>
<thead>
<tr>
<th>Spectrum</th>
<th>Location</th>
<th>Ca/Si</th>
<th>Mg</th>
<th>Al</th>
<th>Si</th>
<th>Cl</th>
<th>K</th>
<th>Ca</th>
<th>Fe</th>
<th>Zr</th>
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<tbody>
<tr>
<td>1</td>
<td>Micro-concretion</td>
<td>1.00</td>
<td>2.64</td>
<td>18.68</td>
<td></td>
<td></td>
<td></td>
<td>18.68</td>
<td></td>
<td></td>
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<tr>
<td>2</td>
<td>Micro-concretion</td>
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<td>1.63</td>
<td>18.11</td>
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</tr>
<tr>
<td>4</td>
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<td>1.80</td>
<td>18.58</td>
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<td></td>
<td></td>
<td>19.89</td>
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<tr>
<td>5</td>
<td>Matrix/clast</td>
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<td>32.88</td>
<td></td>
<td></td>
<td>0.18</td>
<td></td>
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<td>6</td>
<td>Matrix/clast</td>
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<td>0.66</td>
<td>13.73</td>
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<td>4.83</td>
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<td>7</td>
<td>Matrix/clast</td>
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<tr>
<td>8</td>
<td>Matrix/clast</td>
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<td>0.80</td>
<td>7.36</td>
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<td>8.63</td>
<td>2.30</td>
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<td>9</td>
<td>Matrix/clast</td>
<td>0.10</td>
<td>0.89</td>
<td>11.21</td>
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<td>1.14</td>
<td>2.05</td>
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<td>1.02</td>
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<td>8.40</td>
<td>18.65</td>
<td>1.56</td>
<td>3.78</td>
<td>1.15</td>
<td>3.62</td>
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<tr>
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<td>17.68</td>
<td>19.15</td>
<td>1.17</td>
<td></td>
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</tbody>
</table>

Table 2 Elemental Analysis For Each Spectra in Figure 4  (Atomic percentage for each analysed element is given (the balance is oxygen) and the atomic ratio Ca/Si is also provided)
were typically 5-30µm and of quartz (although other minor phases were noted). In addition to primary elements of silica and alumina, the matrix also contained significant calcium, although the proportion was highly variable even over very small distances. For example, spectrum 8 and 9 in Figure 4 are less than 5µm apart, yet the Ca/Si ratio changes from 0.1 to 0.46. Locations from the centre of the clod revealed a very similar situation and Ca/Si ratios were comparable with the matrix from near surface regions.

Thus, it was apparent that the individual clay minerals and any reaction products in these matrix locations were of such small size that they could not be analyzed separately and it appeared that the calcium was closely disseminated in and/or around clay minerals. It is also clear that, other than the micro-concretions, the location/distribution of calcium is similar at locations near surface and deep within the clods, suggesting a common process.

5 DISCUSSION

This study confirms that ion transport can penetrate through clay clods up to 20mm in diameter. This ion transport instigates pozzolanic reactions throughout the clods resulting in a substantial strength increase.

The pozzolanic reactions that drive this strength increase in lime-stabilized soils require further analysis and discussion in context with observations of sample structure.

5.1 Clod Surface Boundary

The application of lime to the surface of large clay clods appears to create a physico-chemical boundary at this point of contact and this is proposed as an important concept for field applications of lime stabilization. The boundary prevented individual clods from joining together to form a continuum during compaction and was maintained throughout all curing periods.

The creation of this boundary may be attributed to the rapid lime-clay reactions operating at the surface of clay particles. The exchange of calcium, for monovalent charge balancing cations instigates a reduction in the diffuse double layer to cause flocculation / aggregation of clay particles [12]. This clay particle rearrangement is typically associated with an immediate increase in the plastic limit [13]. Thus, at these clod surface locations, the moisture content would likely remain substantially less than the increased plastic limit and an air gap between adjacent clods persists.

The implications of a boundary are that each clod would have its own internal system with no significant chemical transfer between adjacent clods. This boundary explains several key observations in this study i.e. inter-clod porosity and lime migration to cause pozzolanic reactions throughout, but not between clods. The preferential concentration of lime into the <10mm clod size group also supports this suggestion (Figure 3). However, other studies [4, 5] have indicated that the external surface of clods continue to receive lime from an external source of lime (described in these studies as a matrix) over long term curing. This does not meet with
the observations of this study which identified that all lime added during mixing was rapidly absorbed into the clay clods and this is considered in detail later.

5.2 Sample Structure Influence

While the clay clods increase in strength with curing, the air voids between clods remain a weakness. Furthermore, although clods are apparently of very low permeability, the sample had somewhat higher transmissivity through the inter-clod void space. Soaked curing of the CBR samples caused a 20% reduction in the CBR value. As soaking did not appear to remove significant calcium (Figure 3), this strength loss appears to have been caused by water ingress at the clod surface and associated softening of the clay minerals. It is also possible that some of this strength reduction was caused by the loss of soil suctions between adjacent clods. In consideration of sample transmissivity, the low surface area of the largest clods appeared to offer the most resilience to strength loss on soaking (Figure 3).

5.3 Mellowing Influences

The application of a 1 hour mellowing period after the addition of quicklime during the first mixing stage of this study appeared to exaggerate strength heterogeneity though the sample. Quicklime slaking generates substantial heat energy, which further accelerates the rate of cation exchange effects and associated increases to the plastic limit [14]. An influence associated with clay mineral aggregations is an increase to the intra-aggregate porosity [15]. This effectively reduces the density of the aggregated region of the clay clod [16]. The mellowing period provided time for Ca2+ ions to diffuse and extend this physico-chemically affected zone deeper into the clod. Energy from subsequent remixing disrupted this relatively brittle layer to form the <10mm fraction. When the material was compacted, the higher strength and reduced ductility of these modified clods resisted remoulding, unlike the larger clods which were little influenced by the quicklime. This caused a localized increase in interclod porosity around these fines (Figure 5).

5.4 Ion Migration/Pozzolanic Reaction

5.4.1 Lime adsorption/gradient

The addition of a lime slurry with the second mix ensured that, irrespective of the concentration of quicklime into the fines, the surface of all clods were coated with lime. This high concentration of lime at the surface of the clods did not cause any problems with the absorption of lime into the clod; after 5 minutes of hand mixing the white lime coating had faded. It is not certain exactly where within the clay-porewater system the lime was stored after this very short time period. The solubility of lime within porewater is very low and would rapidly become supersaturated. While migration / diffusion deeper into the clod was noted over time, this would not be substantial over minutes. Therefore, it is assumed that a process similar to the rapid surface adsorption of Ca2+ ions and hydroxyl groups onto the surface of clay particles [12] facilitated initial storage close to the clod surface. The high surface area of the smectite clay minerals would provide substantial surface area in this regard [17]. Thereby, the storage of both
Ca2+ and hydroxyl groups would instigate ion gradients and the potential for long term diffusion within individual clods [6].

5.4.2 Pozzolanic Reaction Mechanism

The dissociation of lime into hydroxyl groups elevates porewater pH to more than 9-10 at the face of clay minerals resulting in the alkaline hydrolysis of covalent bonds and release of silicates and aluminates into solution [18, 19, 20]. The highly reactive hydroxyl groups combine with these elements to form silanol and aluminol groups, and in the presence of Ca2+ the Al/Si complex will mobilize to form a C-S-H gel [18]. While solubilization of Al and Si may commence at pH 9-10, the efficiency is improved with further increases in pH. Expansive minerals (e.g. smectites) break down rapidly, others (e.g. illite) react at a much slower rate [19]. Therefore, the micro-concretions noted in this study are considered to represent areas where conditions favorable to alkaline hydrolysis were concentrated by the initial rapid adsorption of lime, i.e. at the surface of smectite particles. The dissolution of clay minerals then provided space for supersaturated chemistries within the porewater to precipitate i.e. C-S-H and portlandite.

5.4.3 Diffuse Cementation

The micro-concretions were useful in identifying the pozzolanic reaction mechanism and product composition. However, the relatively minor volume of these features and limited distribution near the clod surface does not explain the significant strength gain observed over the whole clod. It is suspected that, at a sub-micron scale, (i.e. smaller than the resolutions attainable by the SEM used in this study) the C-S-H was also dispersed as gelatinous coatings around aggregations of the clay minerals. The diffuse distribution of this effect throughout a clod would explain its overall strength increase.
A key observation in this study was that the strength throughout individual clods appeared to increase relatively uniformly. Other studies have reported mixed findings in this regard. Davidson et al. [4] described the presence of some soft centers in clods cured for 30 days, although uniform hardness was noted after 90 days. Stocker [5] described a lime diffusion / reaction front, coincident with an improvement in engineering properties, which reached the centre of 20mm clods within 3 days. Therefore, it appears that a migration mechanism can deliver widespread improvements over a clod in a relatively short time period, however, perhaps different soil conditions cause this to slow?

It is suggested that the clay mineral aggregations caused by Ca2+ cation exchange, in combination with saturated inter-aggregate pore water would provide a relatively efficient mechanism encouraging widespread diffuse cementation throughout clay clods [6]. Comparison of Environmental SEM images from the natural clay and the 32-day cure highlight the significance of this aggregation for the present study (Figure 6a and b). The natural clay typically contains individual clay flocss and small agglomerations, coated with thin films of water and separated by air filled pores (Figure 6a). The 32-day cured sample illustrates clay aggregations (e.g. 2-5µm) surrounded and interconnected by pore water / gel-like materials with no visible air gaps (Figure 6b). Much of the clay surface area will remain internal to the 2-5µm aggregations and therefore provides substantially less opportunity for clay surface and inter-aggregate pore water interaction. This reduced tortuosity of pore channels and connected porewater enhances ion migration [6]. Therefore, it is proposed that diffuse cementation is driven by hydroxyl groups migrating via this porewater and instigating alkaline hydrolysis at these locations throughout the clods. The extent of dissolution would be controlled by the concentration and sustained supply of hydroxyl ions, i.e. much less than the extensive dissolution indicated by the near surface micro-concretions. While alkaline hydrolysis consumes OH- and the reaction can ultimately become self-limiting [21] the low surface area from clay aggregation would minimize this in the first instance aiding wider migration. Stocker

![Figure 6. Typical Environmental SEM images obtained from a) the natural clay b) the outer region of a 32-day cure periphery](image)

6 a) Close-up of the natural clay sample  
6 b) Close-up of the periphery of a 32-day cure periphery

Both materials were imaged (before drying) at their natural and cured moisture contents (of 35% and 36% respectively).
[5] suggested such a scenario with Ca(OH)2 reacting on surface contact with montmorillonite. Stocker argued that a single layer of reaction product was deposited, that this essentially blinds the clay particle surface and permits lime to diffuse deeper into the clay clod within only several days curing.

5.4.4 Plasticity Influences

The observed changes to the Atterberg limits (Figure 2) may be linked with the lime-clay reactions noted above. The significant increase in both the plastic and liquid limits by 8 days is consistent with the rapid formation of clay mineral aggregations. In the period between 8 and 194 days further changes in these limits were very small. This suggests that these limits are poor indicators of gelatinous bonding in the inter-aggregate pore space that provides a substantial increase in strength over the same period. It appears that prolonged re-working of samples with pallet knives and application of distilled water is sufficient to disrupt the majority of the gelatinous bonds formed around the clay mineral aggregations. The lower pH and absence of Ca$^{2+}$ in distilled water provides a high potential to solubilize constituent ions from the meta-stable C-S-H gel, facilitating its rapid breakdown [22]. With the dispersion of the gelatinous coating, the underlying clay minerals would be exposed resulting in their usual plastic response. If the reaction product were of greater crystallinity, then a greater influence on plasticity may result. However, this was not apparent with the C-S-H gel formed through lime reaction with this particular clay soil following 194 days cure.

6 CONCLUSIONS

Large clay clods (e.g. 20mm) are a reality for field applications of lime stabilization working with moderate-highly plastic clay. In contrast to findings by previous workers [3], this study has demonstrated that diffuse cementation processes can stabilize these larger clods to provide increased strength and durability following prolonged curing.

A moisture condition wet of the OMC for compaction adds two notable benefits to a lime stabilized soil:

- A saturated pore space between clay mineral aggregations that encourage efficient ion migration and the subsequent rate/extent of diffuse cementation;
- Compaction of clay clods while ductile minimizes inter-clod porosity, reducing this weakness.

It appears that the use of quicklime as an initial treatment, while providing improvement to pulverization, worsens the extent of inter-clod porosity and causes lime to become isolated within fines; away from the majority of the clay soil. This suggests that where the intent of the lime treatment is to achieve the diffuse cementation of moderate – highly plastic clay soils, it may be more suitable for specifications to allow larger clods.
Further studies to confirm the strengthening / weakening mechanisms noted here along with the use of a site relevant preparation method, which includes the use of large clods, would be of benefit.

7 REFERENCES


The identification of micro-chemical effects caused by lime treatment was analysed using scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDX).

Clay clods for analysis were obtained from material within the approximate centre of a soaked CBR sample subjected to a 194-day curing period. The objective of this analysis was to identify at the micro-scale whereabouts calcium, which was added at the periphery, were distributed through the clay clod and whether it occurs as part of a pozzolanic reaction product or in some other form. Total chemical analysis showed that the quantity of calcium within the source clay was at a very low value (Table D.1). This suggests that any increased percentages of calcium observed in the nodules must be due to the added lime.

<table>
<thead>
<tr>
<th></th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
<th>Atomic %</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>40.29</td>
<td>36.87</td>
<td>41.04</td>
<td>Si</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>15.78</td>
<td>14.41</td>
<td>15.97</td>
<td>Al</td>
</tr>
<tr>
<td>Fe₂O₃*</td>
<td>5.26</td>
<td>5.40</td>
<td>4.65</td>
<td>Fe*</td>
</tr>
<tr>
<td>CaO</td>
<td>0.27</td>
<td>0.23</td>
<td>0.26</td>
<td>Ca</td>
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<tr>
<td>MgO</td>
<td>1.27</td>
<td>0.93</td>
<td>1.31</td>
<td>Mg</td>
</tr>
<tr>
<td>SO₃</td>
<td>0.04</td>
<td>0.04</td>
<td>0.04</td>
<td>S</td>
</tr>
<tr>
<td>Na₂O</td>
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<td>0.11</td>
<td>0.15</td>
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</tr>
<tr>
<td>K₂O</td>
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<td>1.68</td>
<td>2.06</td>
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</tr>
<tr>
<td>P₂O₅</td>
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<td>P</td>
</tr>
<tr>
<td>TiO₂</td>
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<td>0.65</td>
<td>0.71</td>
<td>Ti</td>
</tr>
<tr>
<td>Mn₂O₃</td>
<td>0.03</td>
<td>0.01</td>
<td>0.05</td>
<td>Mn</td>
</tr>
<tr>
<td>Cr₂O₃</td>
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<td>Cr</td>
</tr>
<tr>
<td>SrO</td>
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<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>Sr</td>
</tr>
<tr>
<td>ZnO</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>Zn</td>
</tr>
<tr>
<td>Total loss**</td>
<td>33.64</td>
<td>39.11</td>
<td>33.35</td>
<td>Total loss includes 105°C oven dry moisture content and 950°C loss on ignition.</td>
</tr>
<tr>
<td>Sum total</td>
<td>99.49</td>
<td>99.54</td>
<td>99.67</td>
<td></td>
</tr>
</tbody>
</table>

*Iron is assumed to be present as Fe₂O₃.
**Total loss includes 105°C oven dry moisture content and 950°C loss on ignition.

Table D.1 Total chemical analysis of cauldon clay by XRF of fused beads expressed in the percentages of oxides of the three samples and the associated average atomic percentages for the three analyses.

(Analysis courtesy of Joanne Cantwell, Chemist, Lafarge Cement UK)

Preparation
For accurate quantitative micro-analysis using EDX, a highly polished specimen is required and the preparation of these specimens was carried out in accordance with the procedure described in Appendix D of (Winter, 2012). In summary, this involved the careful cutting of clods across the middle with a scalpel, with the resulting half clods then dried at 37°C. Following which they were placed, cut surface down, in a mould, submerged and vacuum impregnated with a low heat, two component resin. Following resin set, the cut surface of the nodule was exposed by abrading the surplus resin with wet and dry sand paper (grade P400).
Further resin was then applied to the flat nodule surface and vacuum impregnated into the pore spaces. After setting, the sample surface was exposed by abrasion of surplus resin with wet and dry sand paper. The surface was then polished, using successively finer grades of diamond suspension, down to a final polish at 0.25µm. At all abrasion/polishing stages isopropanol was used as a coolant and dispersant. The resultant polished samples were carbon coated prior to analysis (Figure D.1a and 1b).

**SEM-EDX Analysis**

The SEM analysis was undertaken in Loughborough University’s Department of Materials, using a Cambridge Stereoscan 360 SEM with Oxford Instruments EDX detector. An accelerating voltage of 20kV was used. Areas from the periphery and core regions from both samples were inspected and data from three general areas from the periphery (P1-P3) and also from the core (C1-C3) of the clods are presented in section 5.7.2.2 (Approximate locations for areas P1-P3 and C1-C3 are indicated in Figure D.1a and D.1b). The elemental composition of individual phases and compounds was determined by spot analysis at each location. Stoichiometric analyses were used to determine the oxygen component in the specimens.

![Figure D.1a (left) and b (right). Cross sections of clay clods from 194-day cured, soaked CBR samples and used for EDX analysis.](image)

**Atomic ratios**

Paper 2 and the main text discusses the atomic ratio Ca/Si and the reason for using atomic ratios in this work is given here. Winter (2012) advises the comparison of atomic ratios to identify hydrated cementitious phases from EDX data, as using individual atomic or weight percentages may be misleading. Cementitious phases and clay minerals possess variable quantities of bound water and/or submicroscopic porosity that cannot be determined using EDX. This results in inconsistent X-ray totals that, in turn, cause fluctuations in the % determination of individual
elements (Winter, 2012). However, the atomic ratios between elements are independent of the analysed total. So, irrespective of changes to porosity and bound water, the atomic ratios would remain constant. The ratio of atomic calcium/silica (Ca/Si) is particularly useful for comparison with values for C-S-H that are discussed in the literature (e.g. (Chen et al., 2004)). Furthermore, in view of the very low value of calcium in the natural clay (Ca/Si = <0.01), changes in Ca/Si are also a viable means of highlighting proportional increases in Ca. This ratio approach was therefore followed for the purpose of this research.
APPENDIX E  NOVEL SWELL TEST PREPARATION

Initial Set Up

A. Measure proctor sample mass. Determine sample volume by measurement with a vernier caliper. Cover proctor sample with a permeable geofabric e.g., terram held in position with loose elastic bands.

B. Assemble CBR mould with perforated base plate and collar. Place a circle of terram on top of the perforated base plate. Arrange proctor sample, stood vertically and centrally within CBR mould.

C. Fill the space between the CBR mould sides and proctor sample with 1-2mm Leighton buzzard sand taking care to maintain the samples in a vertical orientation. Extend this sand layer to approximately 15mm above the top of the proctor sample. To avoid creating excess air voids during this backfill, undertake backfill in 5 even lifts of sand and after each lift continually rotate the CBR mould while striking the outside of the mould with 100 blows of a tamping bar.

D. Insert perforated top plate and use to lightly tamp / level sand near surface. Ensure the top place sits level over the sand backfill (i.e. when measuring from the collar top down to the swell plate (using a vernier calliper) ensure there is no more than 0.5mm difference in depth across the plate diameter). If necessary, adjust sand level and tamp with the top plate until this criteria is met.

E. Adjust top plate screw height until Dial gauge is zeroed. Lower sample into soaking tank/bucket. Re-check zero reading immediately and record any changes.

F. The general arrangement of the completed set-up is shown in figure C.1.

Ongoing Checks / Measurements

G. Adjust tank / bucket water level to ensure the external water level is maintained between 6-10mm over the top plate datum.

H. In first 24 hours take frequent swell readings.

I. In first 2 weeks and after any change to curing temperature, check swell readings daily. Thereafter, record daily on weekdays only.

J. Extra step for bucket samples only. After 35 days at 20°C carefully transfer the bucket (minimizing disturbance to the novel test arrangement within) to an environmental chamber set at 8°C. Take swell readings before, during and after transfer to correct any future readings for plate settlement due to this disturbance.

Swell Test Completion

K. Carefully remove sand and proctor from the CBR mould. Carefully remove terram and gently wipe away any surplus water/sand.

L. Measure sample mass;

M. Measure sample diameter in sufficient locations to estimate the overall sample volume.

N. Measure specimen volume using water displacement method.

O. Dissect specimen to create the following sub samples:

a. All Material from specimen ‘top’ down 20mm;

b. All material from the specimen ‘base’ up 20mm;

c. All material from the specimen ‘sides’ in 20mm;

d. Trim a further 10mm from all sides of the remnant specimen to create a ‘core’ sample.
Figure C1  General arrangement for Novel Swell Test

a, Novel Swell Test Cross section (elevation)

b, Novel Swell Test Cross section (cross section)