

## INFLUENCE OF TEST SETUP ON SULPHATE SWELL MEASUREMENTS

Using low and high sulphate soils to assess swell quantified by different test set ups

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### ABSTRACT

Sulphate swell is a known risk in lime stabilisation and existing literature attributes this to water inundation causing growth of ettringite or thaumasite. Recent studies show sulphate swell is greater at UK site relevant temperatures of 10°C compared to the 20°C often used in laboratory tests and that a mould around samples could limit water ingress.

This study assessed the swell potential of a low and high sulphate soil treated with 3% quicklime. A range of different swell test set ups promoted environments potentially relevant to site scenarios. Two of the swell test methods were based around British Standard methods for soaked California Bearing Ratio (CBR) and accelerated swell tests but using 10°C soaking water and variations saturated with gypsum. A third novel set up (The NTU sulphate swell test) permitted 10°C water to access specimens via a 1-2mm sand surround and including variations with aerated water intended to promote thaumasite growth.

Findings confirm the pathway for water access is highly influential in the degree of swell measured by tests at 10°C, with the most swell measured in the unconfined accelerated swell tests. Adding gypsum to the soaking water caused up to 6.5% expansion in low sulphate samples that otherwise did not swell in tap water, and even higher expansion (22%) in the high sulphate soils. Swell was much less in CBR tests with intermediate amounts in the NTU test. Mineralogy tests indicate only very small amounts of ettringite formed in any specimen and the sulphate swell appears due to unbound water and a potential swell mechanism is discussed.

### INTRODUCTION

A common practice in earthworks for UK construction projects is the treatment of cohesive fill with quicklime. When quicklime is rotavated into cohesive soils, a series of reactions including exothermic drying, cation exchange and pozzolanic reaction occur to result in enhanced engineering properties of the compacted material [1].

The main benefits of using lime stabilisation as a ground improvement method are:

- Improved engineering properties such as high strength, durability, and frost resistance.
- Reduced waste by treating material already on site, rather than removing and importing new material which increases travel to and from site.
- Allowing for wet ground to be dried in winter, therefore allowing earthworks activities to be carried out all year round.

[3]

Although lime stabilisation brings significant benefits, substantial and damaging swell (sulphate swell) has been associated with treatment of sulphate bearing soils, e.g. from gypsum or pyrite [1].

## BACKGROUND

### Sulphate Swell

Sulphate swell is heaving of the treated layers following inundation with water which when sustained can lead to damage of the structures above. Although reports of damage from sulphate swell are relatively rare, when they do occur the additional time and cost to construction projects can be substantial. This form of swell in lime treated layers is associated with the growth of expansive minerals such as ettringite and thaumasite [4]. The chemistry behind this sulphate swell process is summarised further below but in general, swell appears worse under specific environmental conditions e.g. low temperatures and increased water availability [1].

### Sources of Sulphate

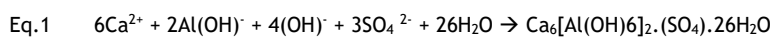
Primary sources of sulphate are found as sulphate salts such as gypsum, which is the most common. In its pure state, gypsum is semi-soluble in water however, its solubility can be four times higher when in the presence of other minerals such as halite (NaCl) [5]. While gypsum has relatively low solubility, fluctuating groundwater to flood the lime treated layer would allow for continuous dissolution of gypsum and growth of new minerals (e.g. ettringite). Hence, an environment which replenishes sulphate ions available for reaction is created.

Secondary sources of sulphate come as a by-product of chemical reactions such as oxidation. The mineral Pyrite ( $\text{FeS}_2$ ) is a common sulphide that is present in soils and when both air and water are available, it oxidises to form sulphuric acid. Where calcium carbonate is also present, a buffering reaction neutralises the sulphuric acid and gypsum is formed as a by-product. Pyrite oxidation to gypsum alone results in at least 35% expansion of the original volume [6] and the resultant gypsum becomes available to form ettringite with further heave potential [7]. Floyd et al. (2003) suggested that environments of either oxygenated groundwater or atmospheric conditions are needed for significant pyrite oxidation prior to formation of ettringite [12]. Soil deposits considered higher risk of sulphate swell often contain a combination of primary and secondary sulphates which should be identified during

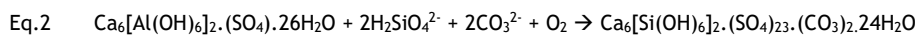
pre-contract laboratory testing, by determination of a total potential sulphate (TPS) value of the soil [8]. A soil TPS of <0.25% is considered minimal swell risk, 0.25% -1% requiring close monitoring with swell tests and >1% of considerable concern [8]. However, specific working methods e.g. including ground granulated blast furnace slag with the lime binder may be used to successfully stabilise soils with TPS up to 2% [8].

### Ettringite and Thaumasite

Ettringite (eq.1) is an expansive mineral which may form from calcium, aluminate and sulphate in an alkaline environment. Ettringite is a hexacosahydrate and where the hydration water is drawn in from an external source a 137% volumetric expansion occurs [6]. Thaumasite has a similar mineralogical structure to ettringite and is described as forming either via ettringite alteration (eq.2), or nucleation on the surface of existing ettringite [9]. Thaumasite growth direct from solution and without ettringite is considered to be extremely slow or unlikely [9].



Calcium + Aluminium Hydroxide + Hydroxide + Sulphate + Water → Ettringite



Ettringite + Silicic Acid + Carbon Dioxide + Oxygen → Thaumasite

Both strength loss and heave are associated with thaumasite formation, however, where this is from ettringite alteration it is noted that water is released and the thaumasite structure will occupy 45% less volume than the ettringite host [10]. The known expansive properties of ettringite and thaumasite have led to their formation being identified as key mechanisms leading to sulphate swell failures [11]. There are specific environmental factors required for ettringite and thaumasite to form. These are as listed and further discussed below:

- Access to water
- Temperature
- Humidity
- High pH

### *Water availability*

The 26 or 24 mols of water in ettringite and thaumasite respectively identifies the minerals as highly hydrous [12], with access to external water a crucial part of their formation as well as transport of sulphate ions through the soil [13][14]. The formation of ettringite and thaumasite often occurs in localised low spots, areas of poor drainage or wet winters [11]. Gypsum has a relatively low solubility in water (2.58 g/L) and is considered to not provide enough sulphate ions to result in ettringite formation. However, where water saturates the soil a mechanism to continuously dissolve and /or transport in the sulphate ions is provided.

### Temperature

The ettringite solubility product mechanism is unchanged across the temperature range 5°C to 75°C, with normal thermodynamic behaviour i.e. reactions approximately double in rate with every 10°C increase and vice versa [15].

Thaumasite requires a <15°C environment to form with an optimal temperature of 5°C with the increasing solubility of carbon dioxide at lower temperatures, i.e. CO<sub>2</sub> solubility approximately doubles as water temperature reduces from 25°C to 0°C, is suggested as the likely cause [16].

According to Hunter [12] and assuming all other reactant availability, it can be summarised:

- > 15°C. Ettringite is stable and will continue to form.
- **Between 10°C-15°C.** Assuming both aluminate and silicate are available then ettringite is kinematically favoured and will form first. However, when only silicate is available then Thaumasite will precipitate.
- < 10 °C. Thaumasite is thermodynamically more stable and will preferentially form. Where this low temperature is sustained for long periods then prior formed ettringite will alter to thaumasite.

### Humidity

Talluri (2013) suggests that humid environments alone do not have enough availability of water for the ettringite reaction [4]. However, a humid environment followed by a saturated environment would allow for the formation of ettringite [4] and this can be expanded upon as follows. As noted above (see sources of sulphate) where sulphate is sourced from pyrite oxidation in the presence of calcium carbonate this necessitates both air and water and so a humid soil atmosphere promotes this reaction. Pyrites in the natural soil may then preferentially oxidise during damp periods of the summer months when the local water table is lower. This would provide a supply of sulphate ions for subsequent formation of ettringite and thaumasite in the winter months, when increased water inflow saturates this previously oxidised zone [17]. This would meet with Talluri's (2013) suggestion that although ettringite forms in a humid environment, the reactions are less common compared to a fully saturated soil. Furthermore, the high pH and humid environment created during the lime stabilisation process i.e. rotovation of lime through the fill while adding mixing water, would encourage rapid oxidation of pyrite to calcium sulphate [1].

### High pH

For a reaction between lime stabilised clay and sulphates in the soil, a high pH is needed [19]. The added lime elevates the pH to 12.4 which increases the solubility of alumina and silica [1]. The high pH also increases the rate at which any sulfide would oxidise to sulphate [20]. Ettringite is stabilised at pH values greater than 12 and becomes less stable at low alkalinity where it will begin to decompose to gypsum [21].

The formation of thaumasite also relies on a high pH of greater than 10.5. The stability of thaumasite is dependent on the pH and once the mineral is formed it can survive

at a pH of 7, although as it gets closer to this value the mineral becomes less stable [13].

#### **Sulphate swell tests within the industry and limitations**

Sulphate swelling measurements have been undertaken measured using a variety of methods in research projects e.g. Beetham's Novel swell test [22] and Abdi and Wild's 3-dimensional swell test developed [23]. However, the two main test methods used in industry, and which are stated as suitable in BS EN 16907-4 (2018) [24] are briefly summarised;

- Soaked California Bearing Ratio (CBR) test with linear swell measurement [25]. The lime treated soil is compacted into a CBR mould, cured unsoaked usually for 3 days (although there is flexibility for different periods) before full immersion 20°C water for at least 4 days. Water passes into the specimen via small holes in base and top metal plates with the linear movement of an unfixed top swell plate recorded. BS EN 16907-4 (2018) requires the test be repeated on at least 3 specimens with failure criteria of average swell >5mm (or 3.94%) or >10mm (or 7.87%) in any single specimen. It is also noted that BS EN 16907-4 (2018) states soaking water should be continuously aerated and soaking should continue for at least 28 days; or until swell ceases. However, these are not requirements of BS EN 13286-47, even in the most recent 2021 revision, and there is no clarity on a method of ensuring water is adequately and consistently aerated.
- Accelerated swell test (AST) [26]. The lime treated soil is compressed into and then immediately removed from a 50 x 50mm mould. The specimen is cured unsoaked at 20°C for 3 days, then wrapped in a permeable geofabric and soaked in 40°C water for 7 days. The average increase in specimen volume of three specimens is reported with typical failure criteria of >5% noted in BS EN 16907-4 (2018).

#### *Sulphate swell test criticisms*

Both standard swell test methods have aspects which may not represent site conditions and therefore could misrepresent the swell potential of a given soil [27].

The tests are performed at temperatures between 20-40°C which does not provide the necessary environment for thaumasite to form. Furthermore, recent studies have shown that when lower soaking water temperatures are used then substantially higher swell occurs. Beetham (2015) reported that reducing the soaking temperature to 8°C after an initial 35 days at 20°C led to a sudden and sustained increase in swell rate of high TPS soils treated with both lime and lime plus GGBS [22]. Newbury et al., compared lime and GGBS treated high TPS soils soaked in 20°C or 10°C reporting 40% more swell in the lower temperature specimens. UK near surface ground conditions are likely to be closer to 10°C and hence the relevance of soaking at 20°C is highly questionable [28].

Further potential issues relevant to both standard tests are the full immersion of specimens in tap water. Full submersion could preclude an oxidising atmosphere for pyrite oxidation and limit the amount of carbonate available to form thaumasite, especially at 20°C where carbon dioxide is less soluble than at lower temperatures. While use of tap water is convenient and consistent, however, it does not account for groundwater saturated sulphate which is a potential field scenario [13].

The CBR test has been specifically criticised for having a steel mould which could; cause adhesion forces that retard swell; restrict water flow into the specimen [17, 29]. The oxidation of pyrite and the subsequent swell is not accounted for in the CBR test. The test only allows for linear swell to be measured which does not represent the swell on site. Conversely, the AST permits fully free access to water with no confinement which could be too severe to reflect field condition?

These criticisms were highlighted by Snedker's (1996) conclusions that the industry requires a more rigorous test that accounts for typical UK ground temperature and allows for saturation and oxidation of pyrite [29].

## **METHODOLOGY**

The purpose of the test programme was to investigate the degree of swell measured with different sulphate swell test set ups. This included using variations on the soaked CBR and AST, as well as an NTU sulphate swell test (NTU test), which was an adaptation of the method developed by Beetham [22] and is further explained below. There were some deviations to the standard AST method (explained later) and so this will be referred to as a modified AST.

The range of environments considered by the programme are presented in Table 1.

### **Soil preparation method**

Two soil types, i.e a high TPS and low TPS soil were used in the investigation:

- Weathered Charmouth Mudstone (part of the Lower Lias group with a high TPS of 2.74% including Oxidisable Sulphate of 0.61%).
- Mercia Mudstone of weathering grade IVa with a TPS of 0.02% and initial consumption of lime value of 1.5%.

3% by dry soil mass of BS EN 14227-11 category 1 quicklime powder (Tarmac Limbase) was mixed into the test soil using a lab approach considered to reasonably reproduce the site process. The required amount of soil was first passed through a 20mm sieve and spread out in a large mixing tray with the lime evenly spread over the surface. The lime was worked in by repeatedly turning over the material for approximately 15 minutes, after which a consistent appearance to the mixture resulted. The mixing tray was then placed inside a very large sample bag which, to create a seal, was wrapped around the tray several times. The tray was then left for a 2-hour mellowing period, after which the bag was removed. The material was remixed with the same method and adding room temperature tap water, using a squeeze bottle to evenly spray over the surface, as necessary to condition the material to a target moisture

condition value (MCV; BS EN 13286-46:2003) of 10.5. An MCV <12 or lower is noted to coincide with a moisture content at or slightly wetter than the optimum moisture content for 2.5kg compaction [30].

**Table 1: The tests and their environments used in the programme.**

Test	Environment		Soil Type	Batch Reference
	Water (10°C)	Confinement		
CBR	Tap Water	Confined	Charmouth Mudstone	CMS_CBR_TAP
	Gypsum saturated tap water		Charmouth Mudstone	CMS_CBR_GYP
AST	Tap Water	Unconfined	Charmouth Mudstone, Mercia Mudstone	CMS_AST_TAP MMS_AST_TAP
	Gypsum saturated tap water		Charmouth Mudstone, Mercia Mudstone	CMS_AST_GYP MMS_AST_GYP
NTU	Tap water Fully immersed	Partially Confined	Charmouth Mudstone	CMS_NTU_F
	Tap water 20mm from base		Charmouth Mudstone	CMS_NTU_B
	Fully immersed and aerated tap water		Charmouth Mudstone	CMS_NTU_A

Following BS EN 13286-2:2012 compaction procedure, all samples were compacted in 3 layers using a mechanical compactor to deliver 2.5kg standard compactive effort. CBRs were compacted into approx. 150mm diameter and 127mm high moulds where they remained for all subsequent curing. NTU and modified AST specimens were compacted into split proctor moulds (approx. 100mm diameter and 115mm high) and samples removed immediately. The standard AST method does require static compression of 50mm x 50mm specimens, however this equipment was not available and so the larger specimen was used. Unsoaked curing was undertaken for 3 days at 20°C with specimens first wrapped in cling film and then sealed in a sample bag.

#### **CBR and modified AST Soaking Arrangements**

The CBR moulds were set up in accordance with BS EN 13286-47:2012. Modified AST specimens were set up in general accordance with BS EN 13286-49:2004 except that the soak periods were extended to 14 days when it was noted the samples were still swelling after 7 days.

For specimens soaked in gypsum saturated water, food grade calcium sulphate dihydrate powder (Saint Gobain superfine white) at a dosage of 4.6 g/l was needed

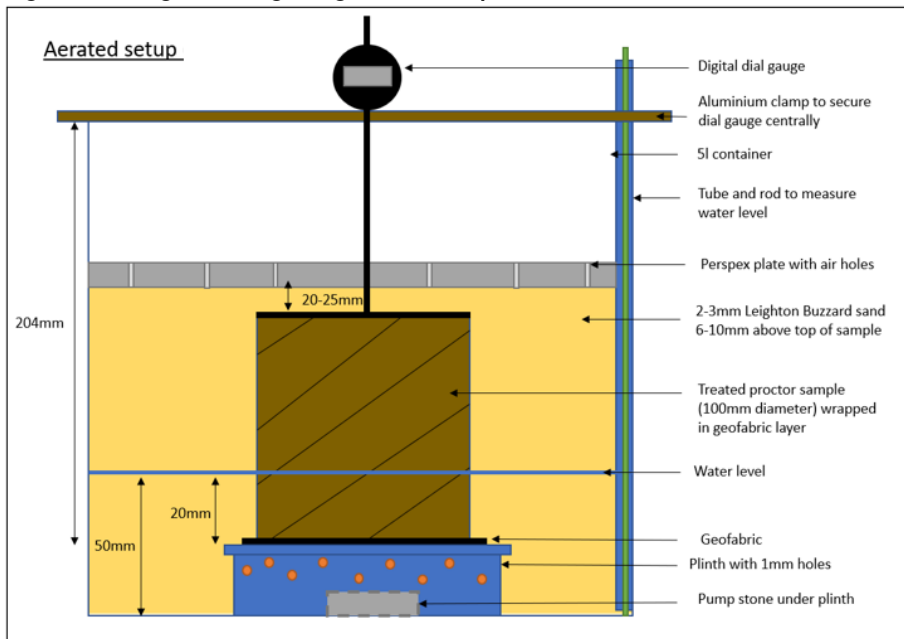
for each soaking, the baths were gently stirred daily to help ensure the water remained saturated with gypsum.

### NTU sulphate swell test

The aim of the NTU test was to introduce the presence of air, in particular CO<sub>2</sub>, to encourage the growth of thaumasite. The NTU test samples were set up in the general arrangement as shown in Figure 1 with 3 soaking water atmosphere variations as summarised in Table 1. After being wrapped in a terram membrane and secured with elastic bands, each sample was placed on a hollow plastic plinth at the bottom of a 5-litre container. The plinth had space to accommodate a porous stone connected via tubing to an aeration pump. With holes drilled into the plinth sides, this provided option to aerate the water. Leighton Buzzard sand was placed around the sample in layers and compacted with a tamping rod. The highly permeable Leighton Buzzard sand (1-2mm) provided confinement to the specimen but allowed for the water to access from all sides of the sample.

The three different NTU test set ups were soaked for 28 days at 10 °C in a fridge with volume on immersion measurements (in accordance with BS EN 13286-49:2004) taken before and after soaking to compare the change in volume of the sample. A swell plate and dial gauge was used to identify when swell occurred.

Figure 1 : Image showing the general set up used for the NTU test.





## RESULTS

### Volume Change % vs Swell Measurements

Figure 2: Graph showing final swell and volume change percentage for the NTU test and soaked CBR.

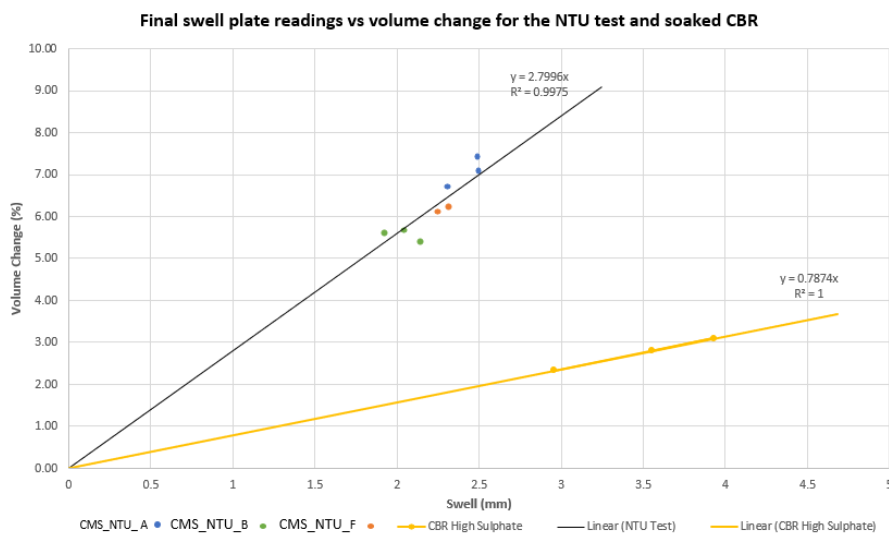


Figure 2 shows the correlation between the final swell plate readings and volume change percentage for the NTU test and CBR test using high sulphate soil. The NTU test had a volume increase of 5.4-7.5% compared to the soaked CBR test which had a volume increase of 2.3-3.1%. This indicates that significant swell is recorded by the NTU test, and this is perhaps related to the easier access to water. This suggests the NTU test is more effective at predicting the degree of risk of sulphate swelling for a scenario where the treated soil is close to the surface and adjacent to regions of high permeability such as a drainage medium.

The coloured dots represent the different test set ups for the NTU test. There is a small variation between the points, however it is not significant enough to conclude that the different test environments cause different amounts of swell.

Accelerated and CBR swell measurements

Figure 3: Graph showing the volume change over time for the AST.

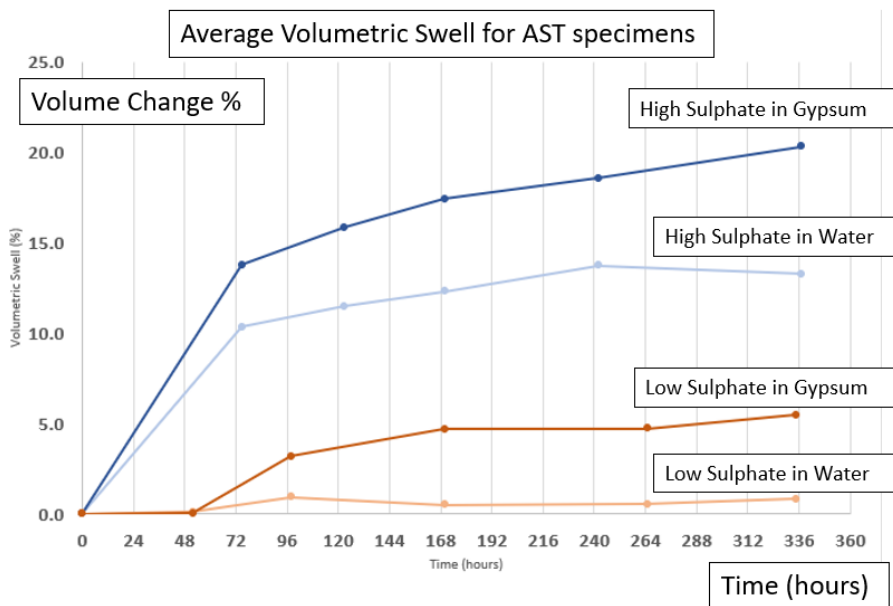
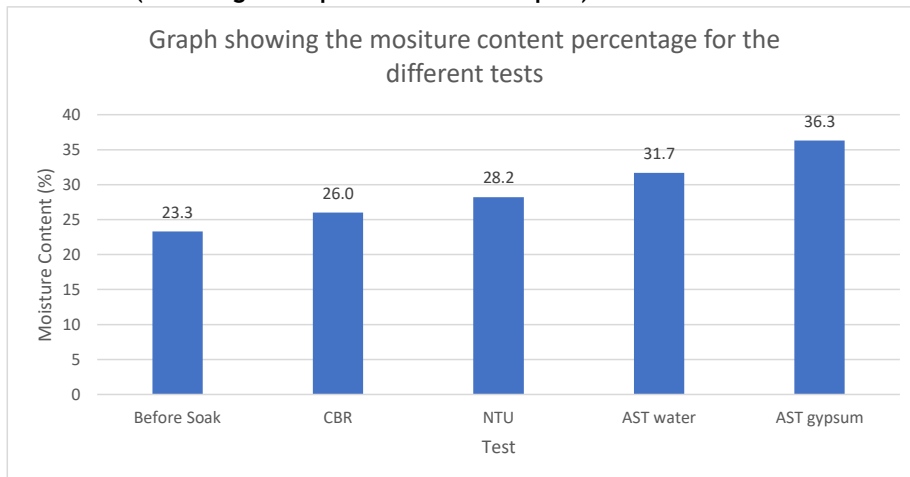


Figure 3 shows the total volumetric swell of the accelerated swell test samples over 14 days. The greatest volume change came from the treated Charmouth Mudstone (high TPS) that had been soaked in the gypsum solution and the final swell measurement was 22%. When comparing this value to the NTU test and CBR, the AST showed significantly higher volume changes and a higher rate of swell. The CBR high sulphate soil samples showed a maximum volume increase of 3.1% which is around a quarter of the volume increase of the same soil mix in the AST samples. This can be attributed to the samples access to water as it can migrate through the samples across their full surface area. The low sulphate soil exhibited minimal amounts of swell after being placed in water. However, the low sulphate soil soaked in gypsum showed an average volume increase of 5% but the swell rate was slower compared to that of the high sulphate soil. This correlates with the results of the high sulphate soil soaked in water as the material swelled by an average of 13%, which is 9% less than that of the same soil mix soaked in gypsum. These results suggest that the introduction of gypsum has had effect on the swell of both the high and low soil samples as sulphate ions have been made available for reaction within the soil samples.

## Moisture Content

**Figure 4: Bar chart showing the average moisture content for the different tests (including the top 20mm of the samples).**

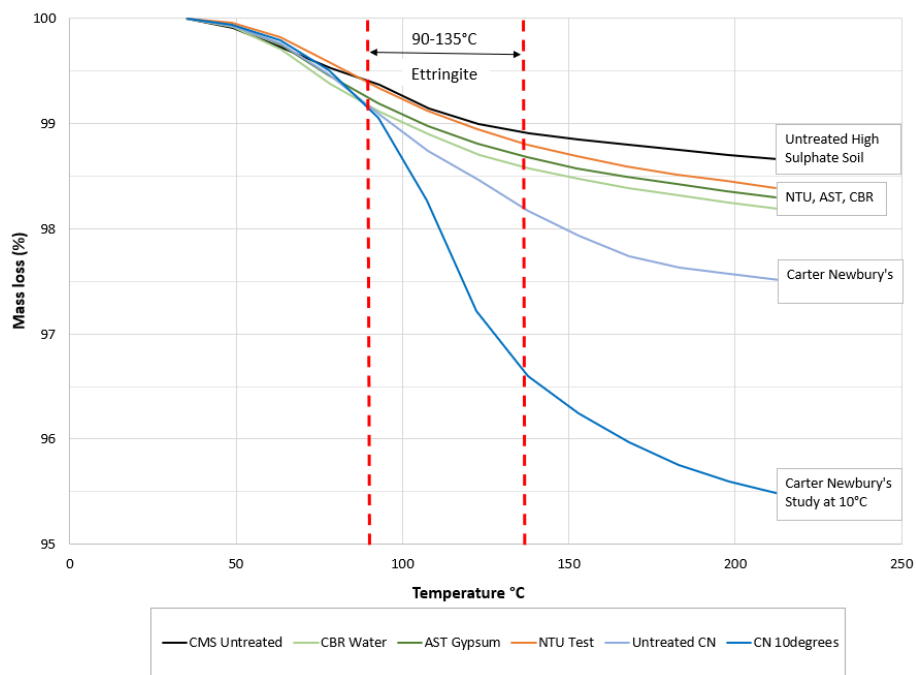


The comparison of the moisture contents of the tests highlights the significance of how the set up restricts or encourages water access into the sample. Figure 4 confirms the expectation that the confined, restrictive samples have a lower moisture content compared to the unconfined samples. On average, the CBR sample had the lowest moisture content proving that the steel mould significantly limits water flow. The NTU test set up allowed 2.2% more moisture into the sample due to water being able to access the whole sample via the permeable sand confinement around the soil. The greatest ingress of water into the samples was found in the AST setups, with the samples soaked in the gypsum-water solution having an average moisture content of 36.3%. This is 4.6% higher than the AST samples that were soaked in only water and 13% higher than the unsoaked soil.

For both the NTU and CBR test, the highest moisture contents were measured at the top 20mm of the samples. This may be due to the potential lack of confinement at the top of the sample which has allowed the top diameter to swell, create cracks and cause water ingress. The AST samples had an even distribution of moisture content throughout the sample, and these were higher than both the NTU and CBR test due to the lack of confinement which allowed water ingress.

## Thermogravimetric Analysis (TGA) Results

Figure 5: Graph showing the temperature at which mass is lost for the samples in the different test set ups.



Thermogravimetric Analysis is a technique used to determine the composition of soils by measuring the mass loss at a range of temperatures. A selection of 40g samples, from the different tests, were analysed over a thermal gradient of 30°C for 10 minutes, then increased to 800°C at a heating range of 10°C/minute, then held at 800°C for 60 minutes.

The expansive mineral ettringite is expected to lose mass between 90-135°C [31], and therefore a soil that contains this mineral should present a significant difference in the gradient of the lines for the untreated and treated soil. Figure 5 shows that all three tests investigated follow a similar gradient and there is only approximately 0.5% mass difference between the untreated and treated soil. This small mass loss suggests that only a limited amount of ettringite had formed. Another significant mineral, thaumasite, is expected to lose mass at 50°C [31], therefore soil samples that contain this mineral should present a different mass loss trend than that shown in Figure 5 between 50-135°C. There is not a clear difference in mass loss between the different tests to suggest that one of the tests produced more ettringite and / or thaumasite than the others.

Figure 5 also includes TGA results from a separate study by Newbury et al., which recorded an average 8.1% volume increase in 10°C soaked CBRs on lime and GGBS treated Oxford clay of 2.51% TPS. When compared to the untreated soil in that study, the Newbury et al TGA data on 8.1% swell specimens identifies an additional 2.5% mass loss between 90-135°C suggesting a significant amount of ettringite had formed.

The comparison between the studies is significant as this study is reporting little indication of substantial ettringite in both CMS and MMS clays, yet the degree of swelling in the tests without restricted access to water i.e. the NTU and modified AST tests were high to very high. This observation on the modified AST tests can be extended further as the low sulphate MMS in tap water did not swell to any appreciable degree. However, when soaked in 10°C gypsum water, swell of around 5% did occur. Across the tests this is suggesting that a sulphate swell mechanism is instigated at this 10°C temperature, but higher swell is not associated with proportionally higher ettringite. Another swell process appears to be at play and as discussed by Beetham [22] this could be due to an ettringite pre-cursor that imbibes water at this low temperature to draw in unbound water but without corresponding growth of crystalline ettringite. This suggestion is tentative and needs more research into swell processes at these site relevant temperatures of 10°C to improve understanding.

## CONCLUSION

The results from this investigation conclude that the test arrangement is highly influential on the degree of swell that occurs. There is a positive linear relationship between the access to water and the volumetric change of the samples. Therefore, the greatest amount of swell was measured in the unconfined accelerated swell test, followed by the NTU test and the least amount of swell in the CBR test. The mineralogy tests indicate that only a limited amount of ettringite formed in the samples and therefore the higher swell is driven by unbound water taken in by the sample. The TGA and X-ray diffraction (XRD) tests (XRD data not presented due to space limitations) also suggest there is no direct evidence of a significant amount of thaumasite growth which was expected at 10°C.

Adding gypsum to the soaking water increases the amount of water taken in by both the low and high sulphate soil samples and therefore increases the volumetric swell. However, the mechanism that encourages this uptake of water has not been identified.

Further research could be conducted on a soil with a higher amount of pyrite to assess the impact of the test environment, in particular the humidity, on the growth of ettringite and thaumasite. This would conclude whether the higher swell is always driven by the access to water or whether the mineralogy plays an equal role. It is clear that the chemical process of sulphate swell at 10°C is not just quantified by the amount of ettringite and thaumasite growth within the soil, therefore further research into these minerals' formation is needed. Also, research is required into the potential ettringite precursors which could account for the high degree of unbound

water being drawn into the soil and leading to the high swell rates at site relevant soaking temperatures. Current standard laboratory tests struggle to identify these mechanisms and cannot replicate all scenarios that could occur on site.

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