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Title	A laboratory study of the expansion of an Irish pyritic mudstone/siltstone fill material
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Publication Date	2012-11-07
Publication Information	Sutton, David, McCabe, Bryan, O'Connell, Aidan, & Cripps, John. (2013). A laboratory study of the expansion of an Irish pyritic mudstone/siltstone fill material. <i>Engineering Geology</i> , 152(1), 194-201. doi: http://dx.doi.org/10.1016/j.enggeo.2012.10.011
Publisher	Elsevier
Link to publisher's version	http://dx.doi.org/10.1016/j.enggeo.2012.10.011
Item record	http://hdl.handle.net/10379/6364
DOI	http://dx.doi.org/10.1016/j.enggeo.2012.10.011

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Cite as:

Sutton, D., McCabe, B.A., O'Connell, A. and Cripps, J.C. (2013) A laboratory study of the expansion of an Irish mudstone/siltstone fill material, *Engineering Geology*, Vol. 152, pp. 194-201.

DOI: [10.1016/j.enggeo.2012.10.011](https://doi.org/10.1016/j.enggeo.2012.10.011)

A laboratory study of the expansion of an Irish pyritic mudstone/siltstone fill material

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Keywords: pyrite, mudstone, normalised expansion, pH, temperature, water

Abstract

Extensive damage to dwellings in the east of Ireland, arising from the expansive pyritic fill material used beneath the floor slabs, prompted the laboratory study presented in this paper to quantify the expansion and study its influencing factors. A novel experimental arrangement, in which sample height and water supply were varied, was used to test a large quantity of active pyritic infill taken from an affected house in the greater Dublin area.

The expansion of ten separate fill specimens was measured over a 175 day period from December 2010 to May 2011. Remarkably consistent results were obtained when the expansions were normalised by the sample height, with expansion rates of approximately 2-3 mm/year/metre height of fill for specimens standing in water, where the rates were largely insensitive to the amount of water supplied. More rapid expansion was noted for one specimen to which no additional water was supplied during the test. A steady reduction in pH of the water, although relatively small, was consistent with pyrite oxidation reactions occurring in the samples during the test, and also implies the occurrence of leaching of ions from the pores in the mudstone and also that oxidation reactions may have been occurring in the saturated mudstone. Changes in expansion were found also to correlate strongly with changes in the ambient temperature in all specimens.

1. Introduction

Pyrite is an iron sulphide mineral that is a commonly-occurring but minor constituent of sedimentary rocks, particularly mudrocks and shales. The mineral occurs in two main forms; cubic crystals that are typically 0.1 – 5mm across and framboids that comprise spherical clusters of minute grains are typically 1- 10µm across. Cubic pyrite, which is colloquially known as fool's gold, is relatively stable in weathering environments, but on the other hand, framboidal pyrite can undergo rapid oxidation in the presence of oxygen and water. Pyrite oxidation is primarily controlled by its surface area; the reactivity increases as the grain size decreases and the surface area increases (Cripps *et al.*, 1993). Acidity liberated by this process reacts with other rock forming minerals or construction materials containing calcium to form gypsum or other secondary minerals, including the iron oxy-hydroxide mineral, limonite. It is likely that pyrite oxidation and gypsum precipitation occur in different environments so there is unlikely to be direct replacement of pyrite with gypsum, but because the unit weight of gypsum is about half that of pyrite there is a net volume increase. In addition, the formation of gypsum crystals on discontinuities within particles and in the void space between particles results in expansion of the fill. Because the enlargement of existing crystals entails less surface energy expenditure than the precipitation of new ones, expansion can occur even if there is void space available within the fill and there is a confining pressure.

Thus, under certain environmental conditions materials containing pyrite are prone to expansion and breakdown over time. In confined conditions such as in underfloor fill material and within hardened concrete, this expansion can lead to significant structural problems, including cracking and heaving of floor slabs. As deterioration of the rock particles in the fill continues, the fractures in the fragments become wider and more numerous, exposing more pyrite and facilitating further oxidation (Evangelou and Zhang, 1995). In effect the reaction becomes a continuous process. Some sources predict that the reaction may continue for more than 40 years (CTQ-M200, 2001, Parfitt *et al.*, 2011), depending upon the amount of pyrite present and the rate at which the oxidation occurs.

Structures have experienced pyritic heave in many parts of the world, including Saudi Arabia, Canada, Australia, Oman, UK, USA, Japan, Norway, and Ireland. Some of these problems have been isolated events, such as the Johnson City Public Library in Tennessee, which experienced remarkable pyritic heave of the library floor (Belgeri and Siegel, 1998). Others have had a more widespread impact; for example approximately 1,000 homes in Japan were

damaged by pyritic heave in 2002 (Yamanaka et al., 2002) and in Canada on-going pyritic heave issues have already impacted upon an estimated 10,000 buildings. In the east of Ireland, it is estimated that between 30,000 to 50,000 homes, most of which were constructed between 1997 and 2007, during the ‘Celtic Tiger’ economic boom years, may be vulnerable to pyritic heave and associated structural problems.

It is very difficult to predict the amount of swelling that will occur in a particular situation. Although maximum volume increase will be limited by the amount of pyrite present, other factors are influential including, as further explained below, the temperature, as well as the amount of calcium carbonate available and the groundwater conditions. Even though pyrite-induced heave has occurred in materials containing as little as 0.1% pyrite (Penner et al., 1972), experience has shown that several years of expansion may be required for damage to manifest itself. Notable damage typically occurs after 3 – 5 years in Ireland and 10 – 15 years in Canada (CTQ-M200, 2001). Several factors have been identified to affect the rate and extent of the expansion, such as depth of the backfill, percentage of pyrite present, density of the fill, water content, position of the water table, grading of the fill, lithology of the rock, and temperature (e.g. Song and Zhang, 2009).

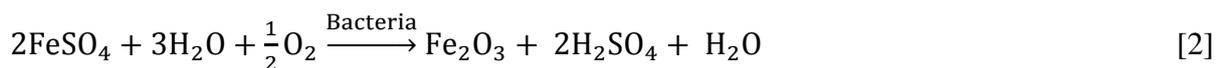
Even though the scale of the pyrite problem in the greater Dublin area is such that the authors anticipate the cost of remediation to run to several billion Euro, very little experimental work has been carried out to investigate this problem. Maher *et al.* (2011) report on a laboratory experiment intended to establish the susceptibility to expansion of an Irish crushed mudstone containing pyrite. The aim of the study, carried out on 600 mm diameter and 300 mm high samples of compacted material, was to develop an accelerated swell test and it was carried out using a number of potentially accelerating factors, including elevated temperatures. However, it was limited in the extent of measuring factors that influence expansion. On the other hand, this paper describes a laboratory study in which the environment of the underfloor fill was simulated. The expansion of ten specimens of the same material was monitored, with measurements of pH to confirm the occurrence of the relevant chemical reactions and consideration of the effect of sample height, water supply and temperature variation within a range typical of the Dublin area.

2. The Pyrite Reaction

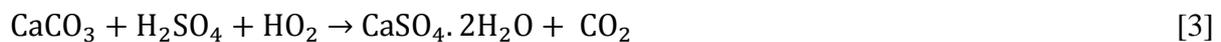
It is generally acknowledged that pyritic heave in confined fill is the consequence of three reactions, simplified versions of which are presented below. Firstly, the pyrite mineral undergoes oxidation of the sulphide, producing sulphuric acid.



In order for this reaction to take place the pyrite mineral must have contact with oxygen and water. Therefore pyritic rock only begins to react when it is exposed to the environment through quarrying or mining. The sulphuric acid produced in reaction [1] will prevent the iron sulphate undergoing further oxidation, as iron sulphate is stable in an acidic environment; however bacteria such as *Thiobacillus Ferroxidans* are capable of converting Fe(II) to Fe(III) in an acidic environment (Hawkins and Pinches, 1997), which results in the formation of the ferric iron oxide, Fe_2O_3 together with sulphuric acid:



When calcium carbonate is present, reaction with sulphuric acid produced in [1] and [2] results in the formation of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and carbon dioxide (CO_2) gas.



The evolution of carbon dioxide gas, which is heavier than air, gives rise to an asphyxiation hazard in confined spaces and excavations. As is the case with most chemical and microbial reactions, the rate of oxidation of pyrite is temperature-dependent (Elberling et al., 2000), although it is generally considered a secondary influence on the reaction. In a remote site in the Arctic, permafrost conditions were utilised to freeze and encapsulate pyritic mine tailings which provided a solution to acid mine drainage problems (Dawson and Morin, 1996, Elberling, 2005). Studies by Ahonen and Tuovinen (1992) showed an almost complete absence of microbial oxidation of Fe(II) at temperatures below 3°C. Meldrum et al. (2001) reported oxidation of pyrite-bearing mine tailings at -2°C, but rates were undetectable at -10°C. It is commonly accepted that Fe(II) oxidising bacteria such as *Thiobacillus Ferroxidans* thrive best at moderate temperatures. Shafikh and Ade (2010) suggest that 36°C is the optimum temperature for them; however this is the subject of some debate, as Hawkins and Pinches (1987) suggest 40°C, Nixon (1978) suggests 30-35°C and Vishniac (1974)

suggests 15-20°C. As the chemical oxidation of pyrite is an exothermic reaction, it tends to create conditions favourable to the metabolism of the bacteria.

The amount of pyrite and its form in the rock, presence of moisture, temperature and the movement of water through the fill all exert some control over rate of and maximum amount of gypsum precipitation. A supply of well oxygenated water to a fractured mudstone which contains pyrite in a framboidal form is liable to lead to a high production of gypsum. The precipitation of this gypsum depends on the solution becoming over-saturated. It is likely that this occurs towards the top of the capillary zone in the fill. In turn the location of gypsum precipitation depends on the grading of the fill and the local groundwater conditions as well as the temperature. The amount of swelling is highly sensitive to subtle changes in the character of the materials and of the environmental conditions. For example, if sulphate derived from a large volume of pyrite bearing mudstone is precipitated in a small volume of fill, then the swelling could be much greater than that produced by the amount implied by the local pyrite content. An additional cause of variation is the habit or crystal shapes developed in the gypsum. Under certain conditions, needle shaped or prismatic crystals that are orientated at right angles to the direction of stress or the surfaces of a crack or cavity in the rock and which incompletely fill the void, are formed. These will cause a much greater swelling than the same amount of tabular gypsum crystals orientated parallel to joints or cracks in the rock.

3. Experimental Testing

3.1 Laboratory Experiment

The laboratory experiment was designed to measure the amount/rate of expansion in simulated *in situ* conditions. A schematic of the apparatus developed to do this is shown in Figure 1a and a photograph of all ten tests in progress is shown in Figure 1b. As indicated, the mudstone was compacted into 225mm diameter medium density 6.5mm thick polyethylene tubes with lengths chosen to accommodate sample heights (H) of 500, 750 and 1000mm (Table 1), where H was defined as the distance from the top of the water to the top of the fill. The tubes were perforated with 10mm diameter holes that were drilled 150mm from the lower end, to facilitate access for water, and 150mm from the upper end to allow access to air.

Pyritic infill material was sourced from beneath the floor slab of a 5-year-old dwelling in north Co. Dublin, Ireland. This material was removed during the remediation of the house which was damaged by expanding pyritic fill material, which in common with other properties in the development, was between 500 mm and 750 mm deep. A maximum floor heave of between 6 mm and 11 mm had been noted for this and similar homes, which had resulted in widespread cracking of the walls and the doors on the ground floor were jamming. However, there was no recorded acidic attack or breakdown in the concrete below floor level. The material was described as a silty sandy fine gravel with angular and sub-angular mainly tabular or platy shaped particles of calcareous mudstone and argillaceous limestone ranging up to about 60mm across. Further data about the chemical and mineralogical characteristics of the fill are given in Section 3.2.

The fill material was manually compacted in layers approximately 200 mm thick using a 200 mm diameter flat based tamper weighing 7 kg which, to ensure the material was not crushed and remained intact, was dropped from a maximum height of 300 mm above the fill surface. As indicated in Table 1, densities of $2000 \pm 50 \text{ kg/m}^3$ were achieved to replicate densities typically found in the underfloor fill. The moisture content of the samples was about 4-5% which corresponds to an air voids ratio of about 10%. The fill was capped with 50mm of compacted sand to provide a flat and even bed for a 3mm thick, 200mm diameter steel plate. The latter provided a flat reference surface for the measurement of vertical displacement (h) relative to the floor on which the samples were placed. This was achieved by mounting an analogue displacement gauge at the centre of each plate. This arrangement meant that vertical expansion was effectively unrestrained and the average vertical displacement was measured.

The samples in their plastic pipes were placed vertically in 45 litre volume clear plastic boxes of size 57 cm \times 39 cm \times 28 cm that contained water of depths (D) respectively of 30 mm, 60 mm and 90 mm (Table 1). A lid was placed on each box to minimise the amount of water lost to evaporation. Standard commercially-available mineral water with an average pH of 7.2 ± 0.1 was used in all experiments. The water level was monitored weekly to ensure the original level was maintained; however it was only necessary to add water to restore the water level once, approximately one week after the experiments commenced. A pH probe and thermometer were used to record the changes in pH and temperature respectively of the water in the plastic boxes.

One experiment (No. 6) was set up with a specimen having H=750 mm and no water (D=0mm) and the programme also included a box with water of depth 30mm as a reference blank to monitor changes in pH of the water in a box without any fill. Some tests on standard pyrite-free Clause 804 fill material were also conducted to measure the effects of self-weight settlement of the fill in the sample tubes. The experiments were performed in two stages; Stage A (specimens 1, 2, 3, 6, 7, 8, 10) followed by Stage B (specimens 4, 5, 9) two months later. The experiments were carried out in an unheated laboratory in order to capture data for a range of temperatures that were more representative of likely ground temperatures than would have been achieved using a heated laboratory.

3.2 Classification of Fill Materials

A visual inspection of a representative sub-sample of the infill showed that two different lithologies were present:

Lithology 1: Dark grey to black, laminated, slightly calcareous mudstone. Clear and white crystal coatings and also some orange-red patches were present on the surfaces of some particles. Most particles were platy in shape.

Lithology 2: Dark grey, calcareous siltstone, some of which was laminated. Some patches of orange-red powdery deposits were present. The particles were mostly blocky and angular in shape.

Particle size distribution analysis indicated that the amount of fines in the infill removed from beneath the floor slab exceeded the Clause 804 limits specified for the project (Figure 2). It is not known whether the fill was non-compliant originally or whether the greater fines resulted from the breakdown of the rock during compaction or due to pyrite oxidation or during excavation and subsequent handling.

It is important to note that laminations in rock would allow the passage of moisture and air through the rock to oxidise the pyrite and facilitate removal of reaction products. The orange-red deposits were probably iron oxide deposits produced by reaction [2], whereas the clear and white crystals were likely to be gypsum, formed by reaction [3] and calcite respectively.

Petrographic examination of a thin section of two fill samples confirmed the presence of calcite and gypsum (Figure 3a) and framboidal pyrite (Figure 3b) in the samples. From

chemical tests for total sulphur, water soluble and acid soluble sulphur, carried out by an accredited commercial laboratory, the amount of pyrite originally present within the underfloor fill was estimated to range from 3.4% to 4.7%. It is widely accepted that a pyrite content greater than 1% is indicative of high swelling potential (CTQ-M200, 2001). Inherent in this estimate is the assumption that all the sulphur now present in the sample was originally in the form of pyrite. Thus the value could be higher if some sulphur has been lost from the system or somewhat lower in the case of a high organic content, as this would contain some sulphur, or if other sulphur compounds were present. It is usual to derive the gypsum content directly from the acid soluble sulphate value, thus making the assumption that no other acid soluble sulphur containing phases are present. Doing this gives a gypsum value of about 2% and by subtraction of the acid soluble sulphur value from the total sulphur value, the derived equivalent amount of pyrite now present is calculated to be between 2.1% and 3.6%. This implies that between two thirds and three quarters of the original was still present to react, and may in the future become oxidised. The total sulphur minus the acid soluble sulphur (present as sulphate) for the samples was between 1.1% and 1.9%, which is higher than the threshold level of 0.5% suggested by Hawkins and Pinches (1992) to indicate an aggregate with a potential for swelling. This potential for swelling is due to the presence of unreacted pyrite in the material, although for various reasons it does not mean that swelling is inevitable. The data derived from the examination of thin sections of the rock using optical microscopy tell a similar story, although the estimated amount of pyrite present in the rock was around 1.5 – 2%; however, as it is not possible to discern very fine-grained particles using this method, this is liable to be an under-estimate of the pyrite content. It should be noted that this assessment is based on the analysis of samples taken from only two locations within the mass of infill beneath the house. These samples are believed to be representative of the fill but care should be taken with extrapolation of the interpretation presented in this study to other materials as variations are likely to exist in the material used throughout the construction due to compositional variations typical of geological materials. Further studies are necessary to investigate the impacts of this variation on the expansion behaviour.

4. Results

The experiments were conducted over a 175 day period from December 2010 to May 2011. The vertical displacements of the top-plates (h) have been expressed as a normalised

expansion (h/H) for subsequent interpretation. The specimens displayed a period of net settlement for approximately 21 days. This was attributed to self-weight settlement and/or some loss of moisture from the fill above the capillary fringe and sand capping being greater in magnitude than any expansion that occurred. It may also have been caused by softening of the rock at points of contact with water, which would lead to self-weight settlement of the fill. Only the data beyond 28 days, once a steady expansion rate was established, are used in subsequent plots. The tests on standard pyrite-free Clause 804 fill material were used to confirm the length of the self-weight settlement period. The normalised expansions (h/H) for Stage A and Stage B experiments are shown in Figures 4a and 4b respectively. The data are remarkably consistent, and do not show any apparent relationship with density over the small range of densities used in the tests (shown in Table 1).

4.1. pH levels

The pH values initially increased during the first 2 weeks in all experiments. These increases in pH were highest in specimens with lowest water depth D and least dilution capacity. Experiments with the same water depth exhibited very similar initial rises in pH; by way of example, the $D=30\text{mm}$ case is shown in Figure 5 in which a pH a rise of 0.9 ± 0.1 occurs. Interestingly, a rise in pH of 0.5 was recorded in the water of the box without any fill material (reference blank) within the first ten days. This may be due to the loss of CO_2 from the water. The difference of 0.4 was possibly due to the slightly alkaline nature of the original mudstone.

Once this initial period of increasing pH was complete the pH levels began gradually to fall in all the experiments where mudstone was present (Figure 5), suggesting that the pyrite in the fill material was undergoing oxidation and producing sulphuric acid. This suggests that leaching of ions from the unsaturated mudstone into the water was occurring, possibly as well as oxidation of pyrite in the saturated mudstone. The steady decline in pH mirrors the steady continuous expansion evident in Figures 4a and 4b. A plot of the change in the recorded pH (after the initial rise) against H confirms that the amount of sulphuric acid produced is in proportion to the quantity of fill material (Figure 6).

4.2 Influence of temperature

The temperature of the water in the boxes is superimposed onto the normalised expansion versus time graphs in Figure 4a (Stage A experiments), and a closer examination reveals that

the expansion correlates strongly with temperatures in the range of 4⁰C -15⁰C. A zoomed-in plot (75-115 days, during which time the temperature varied from 7.9⁰C to 11.3⁰C) is provided in Figure 7 for clarity. It can be observed from Figure 7 that a major drop in temperature results in a drop in the rate of expansion. This is what would be expected given that a drop in temperature will reduce the evaporation of water and therefore reduce the rate of gypsum precipitation. It is likely that a reduction in the rate of pyrite oxidation also occurred. In some cases a period of low temperature results in a delay in expansion for some days. This is most likely because the bacteria suffered the effects of thermal shock. This is a common occurrence in mesophilic bacteria that grow best in non-extreme temperature conditions as a very low temperature would cause mortalities. This would then be followed by a gradual recovery of the population once the temperature ameliorates. Tests by Allen (1923) on mesophilic bacteria at temperatures a little above their thermal death point, confirmed a reduction in their activity due to temperature shock.

The effects of thermal expansion of the fill and of the steel members to which the displacement gauges were attached were not specifically determined in these experiments. However, using an upper limit published value (e.g. Trautwein-Bruns et al., 2010) for the maximum coefficient of thermal expansion for mudstone ($8.5 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$), for the temperature ranges experienced, the maximum thermal expansion would be likely to be of the order of 10-15% of the expansions recorded in these tests. Changes to the temperature of the steel work were calculated to have a negligible effect on the dial gauge readings.

The differences in the timing of pyrite-induced damage experienced in Ireland and Canada may be due to climatic effects. Notable expansion has been observed to occur after 3-5 years in Dublin (where the average temperature is about 10⁰C and there are longer periods of warm weather and no months in which average temperatures are below 0⁰C) and 10-15 years in Quebec (CTQ-M200, 2001) (where the average temperature is about 4⁰C and for about 5 months of the year freezing average temperatures occur, which would inhibit bacterial action and ground water movement). Floor level survey records suggest that unoccupied houses from the Dublin area that have never been heated internally (and therefore have a lower thermal gradient between the fill and the internal air than occupied houses) register slower heave than occupied homes.

4.3 Water supply

The variation in water depth (D) in the experiments (where water was supplied) was found to have minimal influence on expansion rates (Figure 8). Interestingly, in experiment 6 (H=750mm, D=0mm) with no water other than that already present, expansion took place at a faster rate than in the other experiments. According to Hammack *et al.* (1990), inundating pyritic material could reduce oxidation rates by at least 96%, although this will depend on the composition and rate of water movement. This reduction in expansion rate due to inundation is probably because atmospheric oxygen is excluded and by dilution raising the pH so the conditions become less suitable for the bacteria. Jerz and Rimstidt (2004) have also shown that oxidation rates in moist air are significantly faster than those for aqueous pyrite oxidation. Results of this study suggest that the use of drainage to limit the availability of water to the fill may not be successful at slowing or preventing swelling but could even increase it; however it is possible that less expansion was recorded in the samples to which water was supplied because the saturated mudstone was softened by the presence of water resulting in self weight settlement. The impact of these factors on the measurements would be to reduce the observed expansion. Further work is required for the role of the presence of water on the expansion process to be fully comprehended.

The results of Experiment 6 demonstrate that the water already present within the sample and is available from the atmosphere in the (unheated) laboratory was probably sufficient for pyrite reaction to occur as it is unlikely that there was sufficient gypsum in solution to account for the observed volume of swelling. It is not clear what the effect of a lack of a water table would be in the long-term but in the absence of water, movement of reaction products would cease, so it is likely that at some point expansion would be arrested. With a continued supply of water, it is thought that the expansion process would continue until all the accessible pyrite or calcite had been consumed.

4.4 Expansion predictions

It is evident from Figure 8 that, for the specimens with a water supply, the expansion rate is proportional to the height (H) of the fill. An average expansion rate of about 2-3mm/year/m height of fill is implied by the data in Figure 8, and although it is acknowledged that the data did not pertain to a full annual cycle, it has captured a half-cycle which is offset by 1 month or less from the corresponding half-cycle between the coldest and warmest periods of the Irish climate. When this rate is extrapolated to the field scale (with approximate correction

for thermal expansion of the mudstone), for houses with 0.5 m -0.75 m thickness of fill in place for 5 years, the estimated expansion is about 6 mm -9 mm, which is consistent with the aforementioned 6 mm -11 mm deduced from floor slab level surveys.

The effect of surcharge pressure on the amount and timing of expansion was not investigated in this study. However, Taylor and Cripps (1984) report that in the literature they review maximum heave pressures values of the order of 0.5MPa are quoted. This is comparable to the pressure created by a typical floor slab, whereas theoretically values much higher than this are feasible. Observations of prismatic gypsum crystals formed in incompletely filled cracks indicates that widening occurs against confining pressures and causes expansion in excess of the volume of gypsum precipitated. Expansion would tend to compress the fill, leading to an increase in density and modulus, and would also result in compression of the polystyrene insulation placed beneath the floor slabs. However, no measurements of these effects were made.

5. Conclusions

The results of an experiment in which the expansion of a pyritic fill in simulated underfloor conditions is modelled have been presented in this paper. The following conclusions have been drawn:

- (i) A slow and steady decline in pH readings measured in the water in which the samples were placed, suggests that pyrite oxidation was resulting in the formation of sulphuric acid in the interstices of the mudstone. Reactions in the saturated mudstone are not precluded, but it is most probable that oxidation would occur mainly in the unsaturated mudstone above the water table, which implies some leaching of ions from the porewater at the water table. This notion is supported by the finding that the reduction in pH is proportional to the depth of pyritic rock fill used in the experiments.
- (ii) The results of the experiment also proved that a definite relationship between exists between temperature and expansion rate over a temperature range liable to be experienced beneath buildings in Ireland. The swelling measured was significantly greater than would be expected due to thermal expansion of the mudstone and steel. Previously temperature had been considered a minor factor affecting the rate of

expansion. The difference in average annual temperature is put forward as a possible explanation for why pyrite causes damage in homes much sooner in Ireland than in Canada, for example, although as well as climatic effects, differences in construction methods, groundwater conditions and the mudstone may also be influential.

- (iii) The variation in the water supply at the base of the experiments has been shown to have little effect. However the experiment with no water displayed expansion at a greater rate than those with water, providing evidence that saturation may hamper expansion and residual moisture and atmospheric humidity are sufficient to allow the pyrite reaction to take place, at least for the duration of this experiment. However, it is possible that softening of the mudstone particles due to contact with water, may have reduced the amount of swelling observed in these tests.
- (iv) The laboratory-measured expansion rates were found to vary approximately linearly with fill thickness and these rates are consistent with the amounts of expansion typically measured in dwellings in the greater Dublin area. The success of the experiments was deemed to be due to the fact that the fill was active at the time of sampling and the experimental setup was representative of the field problem being considered.
- (v) Further research is required to determine the effects of variation in the pyrite content of the mudstone on the rate and amount of swelling. It is expected that current work, including modelling of the effects of mineralogical changes on the behaviour of the fill and the performance of the floor slab and supported structure, will facilitate prediction of the effects, if any, of variations in these factors. Research is also required to determine the impact of variation in the grading of the fill and its state of the compaction on both the magnitude of heave pressures and the effects of physical and mineralogical changes on the geotechnical properties of the fill.

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Test ID	Sample Height, H (mm)	Water Depth, D (mm)	Pipe Length (including 50mm sand) (mm)	Density (kg/m ³)	Stage
1	500	30	580	2043	A
2	500	60	610	2050	A
3	500	90	640	1963	A
4	750	30	830	1959	B
5	750	60	860	1955	B
6	750	0	800	1949	A
7	750	90	890	2005	A
8	1000	30	1080	1944	A
9	1000	60	1110	1950	B
10	1000	90	1140	1940	A

Moisture content 4-5%, and air voids ratio 10% approx.

Table 1. Sample heights, water depths, and densities used in experiments.

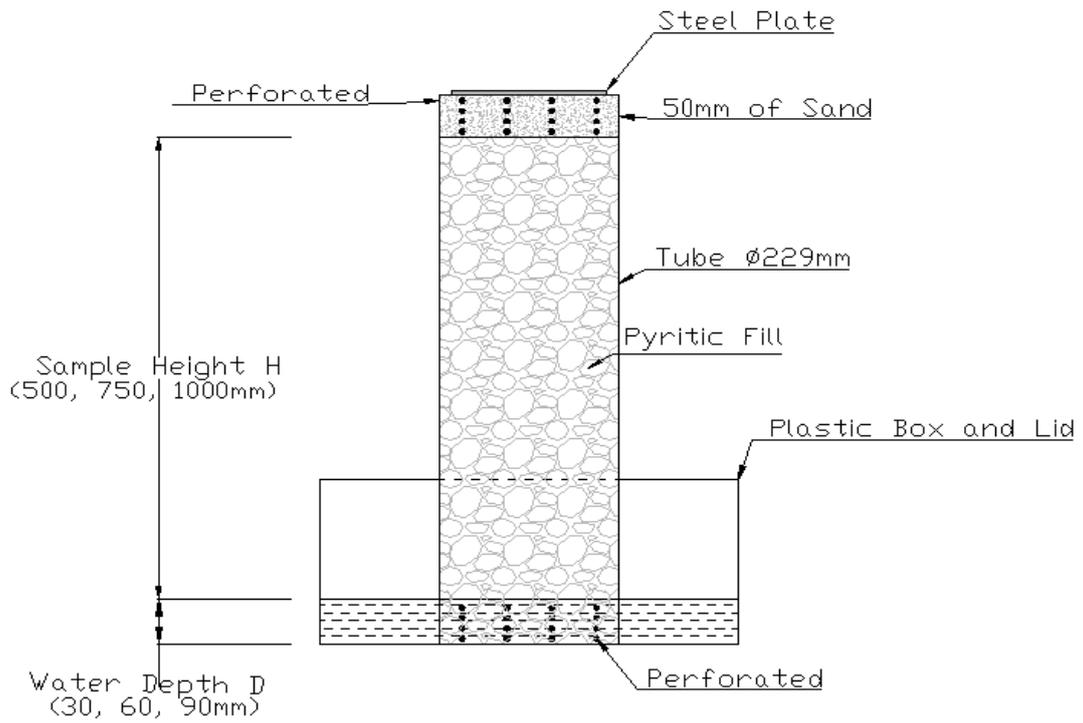


Figure 1a: Schematic of experimental apparatus (Values of H and D are given in Table 1).



Figure 1b: Test specimens situated in unheated laboratory.

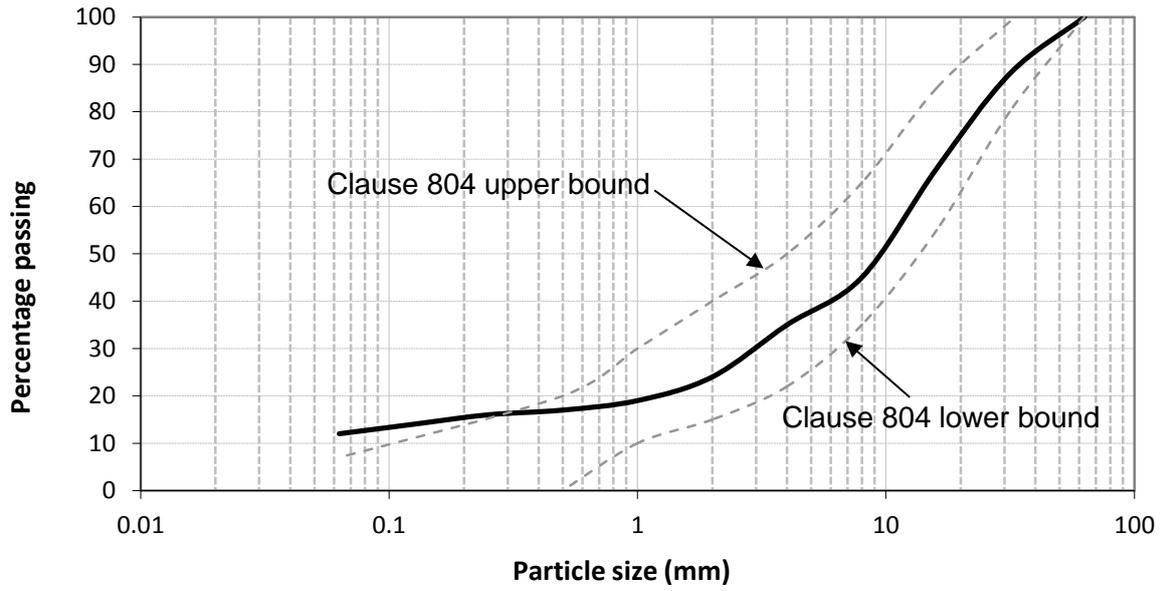


Figure 2: Particle size distribution of the rock used in the experiment compared with Clause 804 specification.

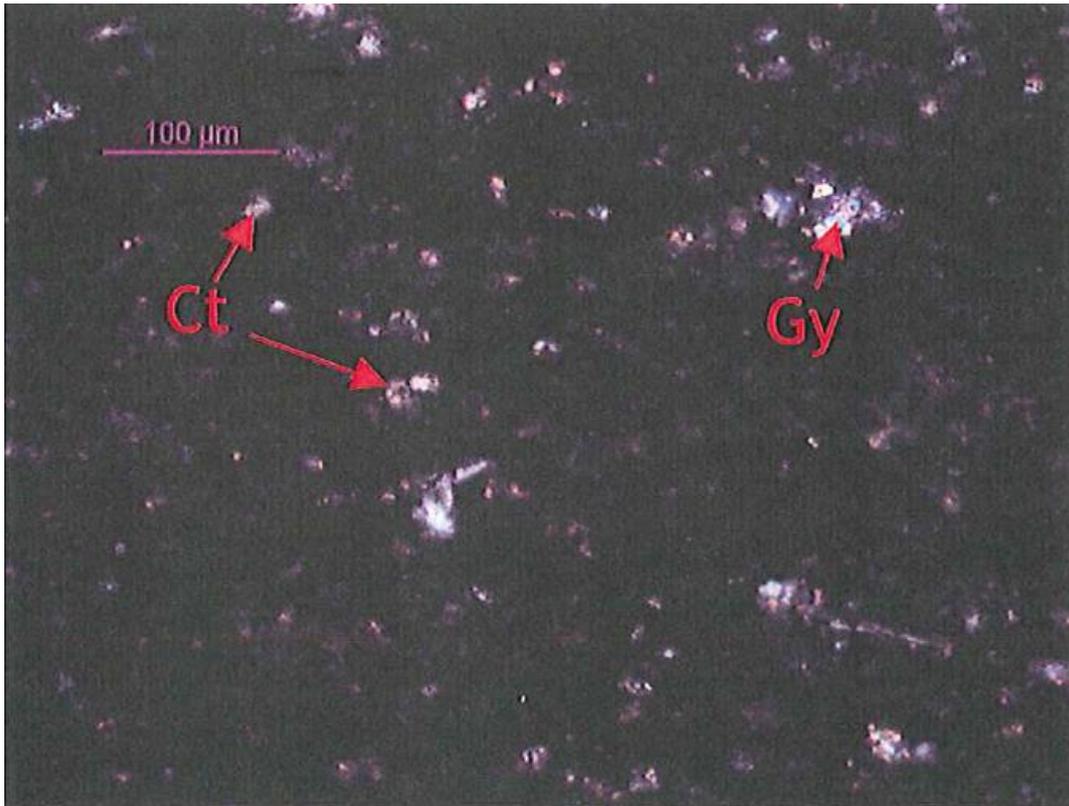


Figure 3a: Photomicrograph: typical composition and structure of calcareous mudstone in fill, showing gypsum (Gy) and calcite (Ct). Note 100μm scale bar.

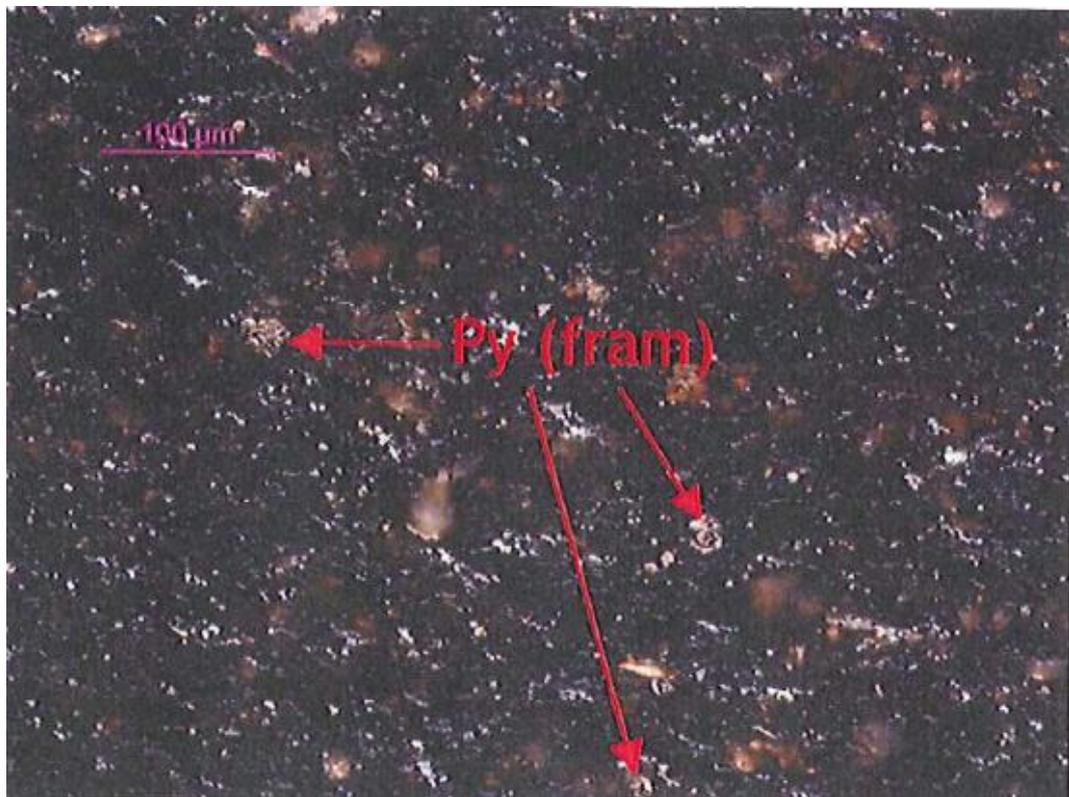


Figure 3b: Photomicrograph: typical composition and structure of calcareous mudstone in fill, showing framboidal pyrite (Py). Note 100μm scale bar

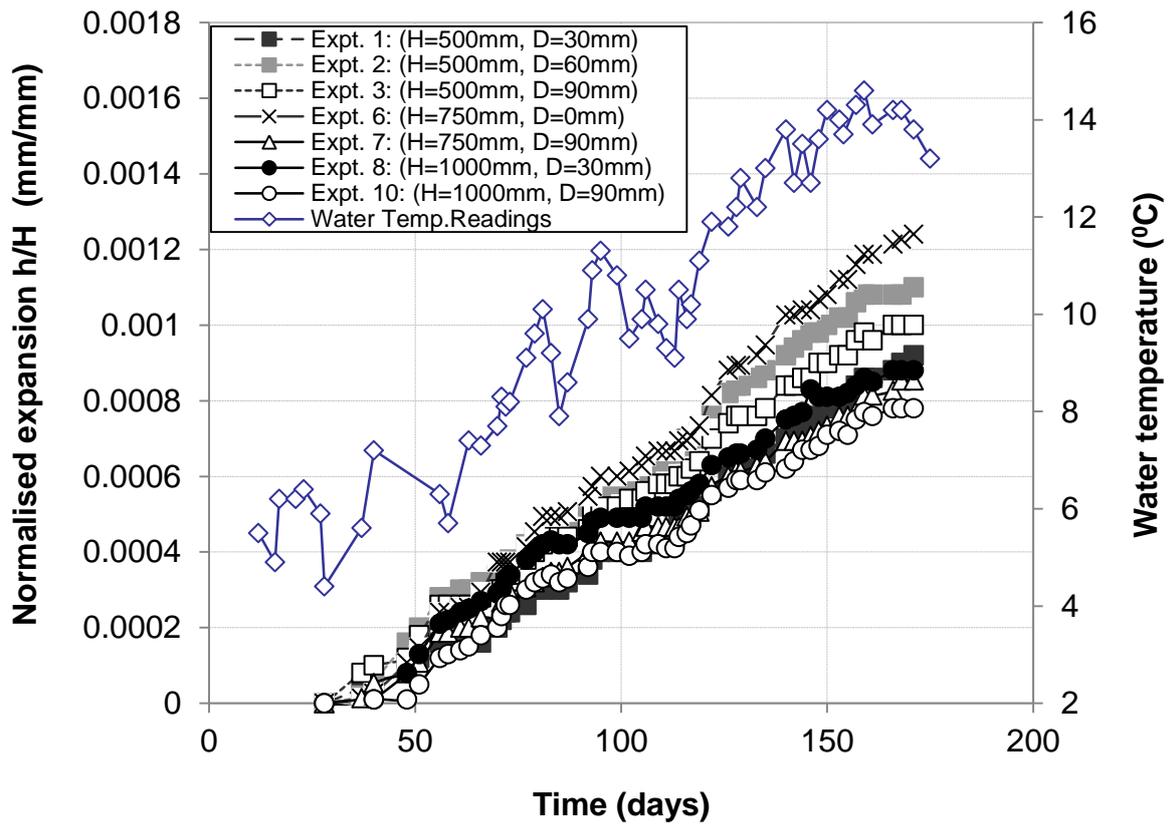


Figure 4a. Normalised expansion for Stage A experiments (where H and D are defined in Figure 1a)

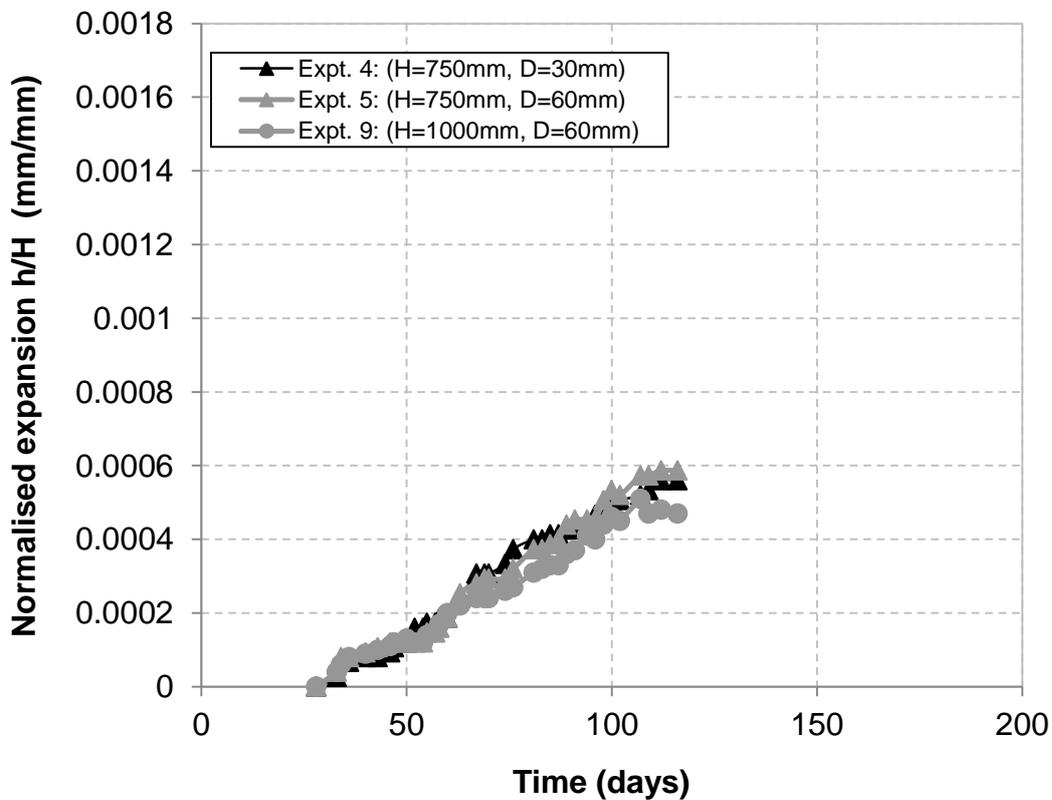


Figure 4b. Normalised expansion for Stage B experiments (where H and D are defined in Figure 1a).

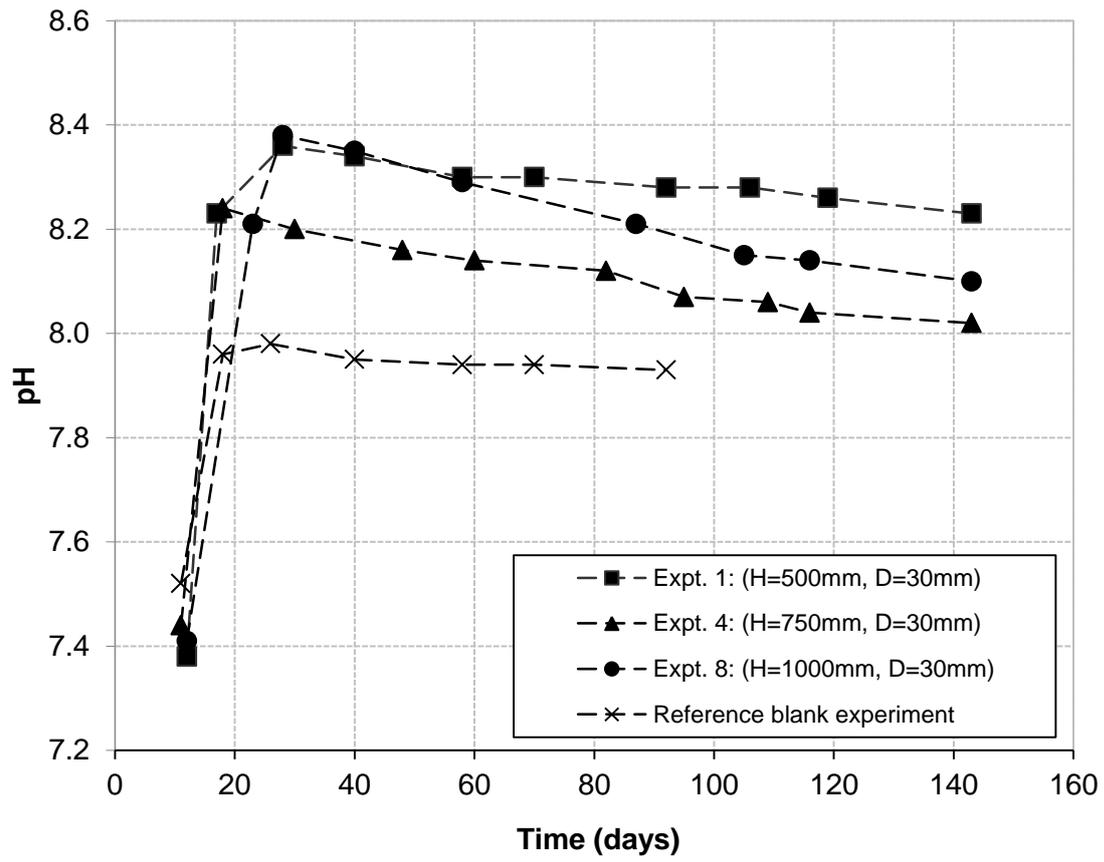


Figure 5. pH-time variation for experiments with D=30mm.

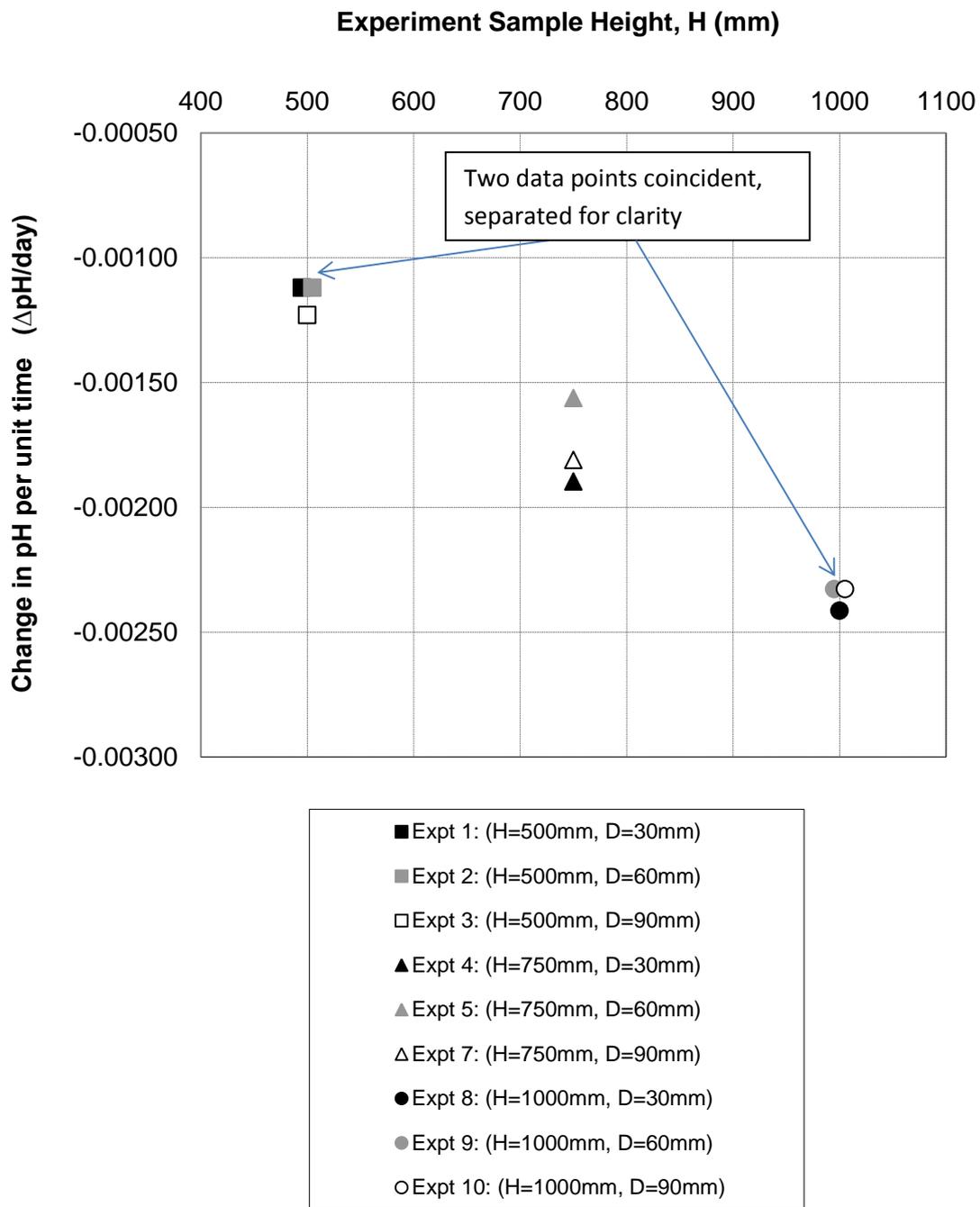


Figure 6. Change in pH versus amount of fill (sample height, H) above level of water.

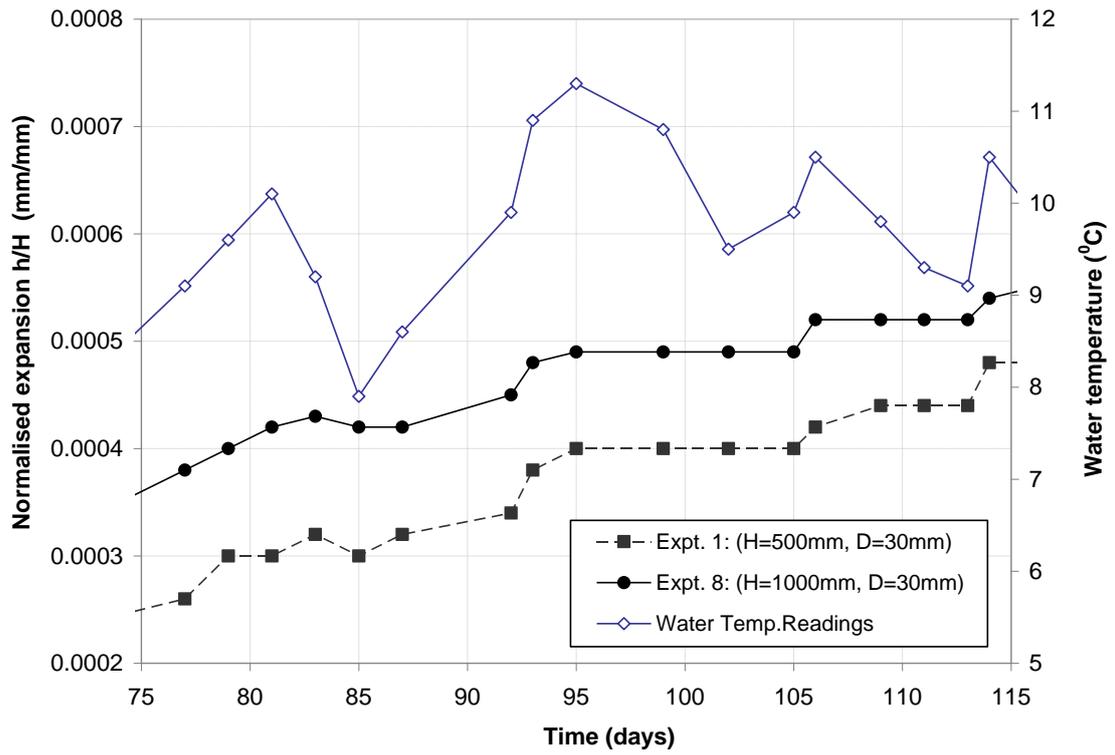


Figure 7: A zoomed-in view of the normalized expansion-time plots for experiments 1 and 8.

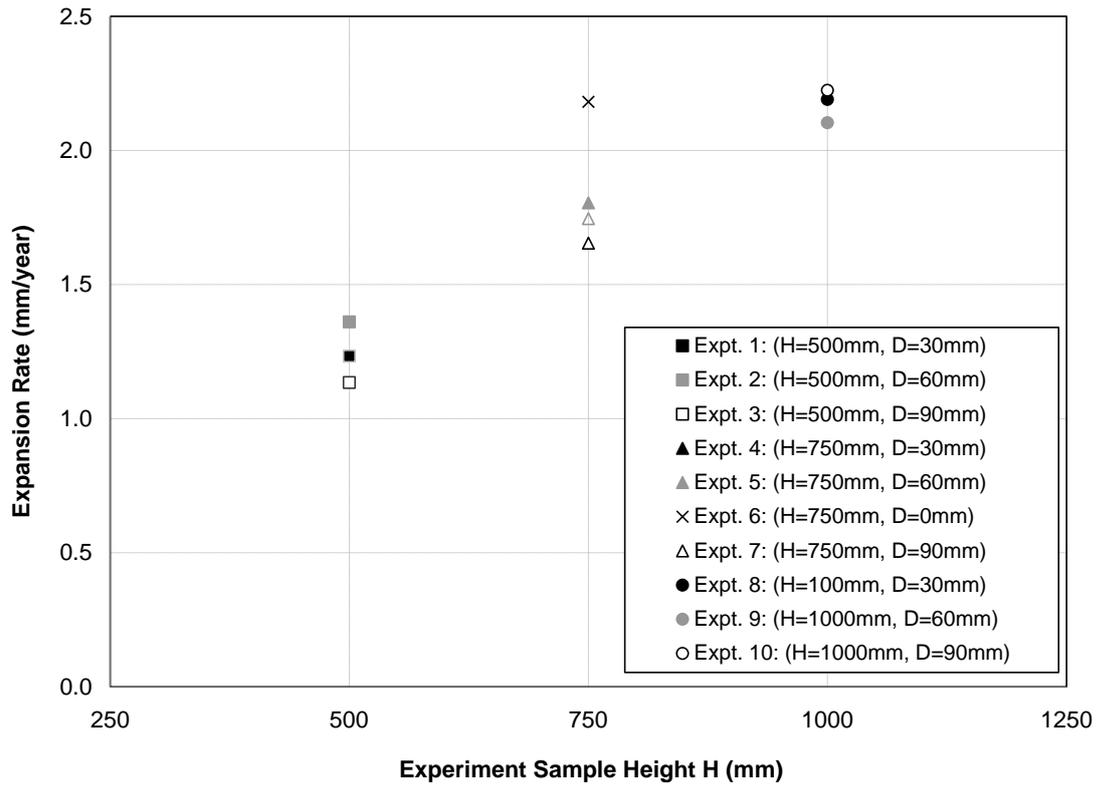


Figure 8: Expansion rate versus amount of fill above water level