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Pyritiferous mudstone-siltstone: expansion rate measurement and prediction

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Abstract:

The expansion rates of a pyritiferous Irish mudstone/siltstone fill material have been measured over a period of 19 months in an apparatus devised to replicate underfloor conditions. The testing, performed in a temperature-controlled environment, has shown that both fill density and depth submerged in water have significant influences on the progress of the expansion. It appears that while thermal expansion/contraction has an effect on expansion rates immediately after a temperature change, there is no long term temperature effect on the rate of expansion. In addition, an examination of chemical test results for 60 houses in a housing development in the Dublin area has confirmed that pyrite content is the dominant control over the degree of expansion. A molecular/molar analysis of the pyrite chemical process equations, in addition to a knowledge of the original pyrite content and rate of oxidation, has been used to give a lower-bound estimate of the amount of heave in the laboratory experiments.

The economy of the Republic of Ireland experienced a period of unprecedented growth between approximately 1995 and 2007, known as the ‘Celtic Tiger’ years. The construction sector contributed significantly to this growth; for example, in 2006 the building and construction sector is estimated to have accounted for 9% of GDP and 10.4% of GNP, 64.3% of which was fuelled by house-building (CSO 2008). In the same year, the number of housing units completed was 88 188 (compared to the 1995 figure of 30 575, CSO 2008), which amounted to approximately 50% of that in the neighbouring U.K., a country with a population approximately 15 times that of the Republic of Ireland (Barrett *et al.*, 2007).

This escalation in residential construction prompted an unprecedented demand for fill/hardcore material, especially in the greater Dublin area. The Report of the Pyrite Panel (Tuohy *et al.*, 2012), commissioned by the Minister for Environment, Community and Local Government, citing the Irish Concrete Federation, notes that the estimated volume of quarried stone products in Ireland increased by almost 250% between 1993 and 2007. Some of the material quarried contained pyrite, an iron sulphide (FeS_2) mineral that is a commonly-occurring albeit minor constituent of sedimentary rocks, particularly mudstones and shales. Pyritiferous materials are prone to breakdown over time in certain environmental conditions and when confined such as under floors or in concrete, due to the precipitation of expansive minerals, significant structural damage may occur, including cracking and heaving of floor slabs. For example, Hawkins and Stevens (2014) document in detail the Ballymun Youth Facility in Dublin where the use of pyritiferous fill led to structural damage and required remediation. The pyrite reaction process and some governing factors have been described by Reid *et al.* (2005), Sutton *et al.* (2013) and others.

Tuohy *et al.* (2012) note that the pyrite problem was largely unknown to design professionals and the Irish construction sector prior to 2007. This is in spite of instances of damage induced by pyritic heave being reported internationally, notably in Canada, the U.S.A. and the U.K. Damage to residential buildings in Canada due to pyritiferous fill was reported by Ballivy *et al.* (2002) and others while Hawkins and Pinches (1987) report on damage due to underlying pyritiferous bedrock at Llandough Hospital in Cardiff where construction began in 1927. The Irish problem presented itself in a much shorter period than in Canada, possibly due to some combination of the parent geology, pyrite type, moisture content, construction methodology and climate.

In March 2012, 74 separate housing estates comprising 12,250 ground floor dwellings with potential for suffering pyritiferous heave problems were made known to the Pyrite Panel. Approximately 1,100 on 12 estates had already been remediated or were in the process of being remediated, and owners of \approx 850 dwellings had registered a claim with a guarantee provider. The Panel estimated conservatively that there may be approximately 10,300 more dwellings with pyrite present in the hardcore, although not all of these dwellings will necessarily be affected by heave. Their report also estimated the average cost of remediation to be in the region of €45,000 per house. Work by two expert subcommittees in conjunction with the National Standards Authority of Ireland (NSAI) has led to the launch in early 2013 of IS 398, a new Irish standard dealing with the inspection/testing (Part 1) and remediation (Part 2) of properties damaged by pyritic fill heave.

In parallel, experimental research has been instigated nationally to quantify the expansion rates in active pyritiferous material. Maher *et al.* (2011) report a laboratory experiment intended to establish the susceptibility to expansion of an Irish crushed mudstone containing pyrite. Accelerated test conditions were imposed upon the 600mm diameter and 300mm high samples and the reaction showed no sign of abating after 2 years. Sutton *et al.* (2013) developed the novel experimental arrangement shown in Figure 1 to simulate the environment of underfloor fill. With pH measurements to confirm the progress of the pyrite reaction in a mudstone/siltstone material, the authors demonstrated a proportional relationship between specimen height and amount of expansion over a 6 month period and normalised expansion rates were consistent with those typically measured on heaved floor slabs in the greater Dublin area. The expansion rate was reported to respond quickly to changes in ambient temperature (in the range 4°C to 15°C approx. in an unheated laboratory) while the thickness of samples submerged in water was found to have little effect, although interestingly, a specimen without access to water other than the natural moisture at the time of compaction expanded the most.

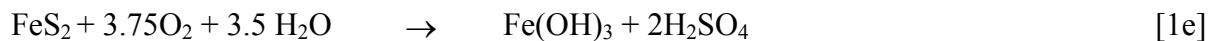
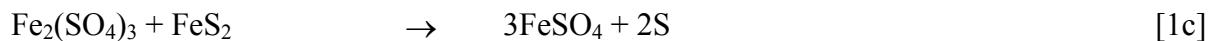
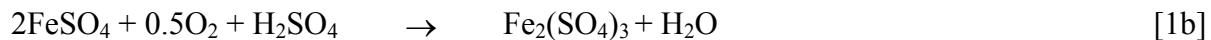
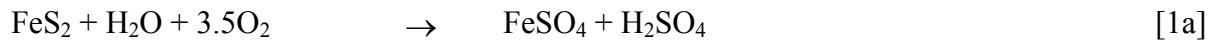
In this paper, further test results using the Sutton *et al.* (2013) apparatus are presented in which the effect of temperature, fill density and water supply have been studied more systematically (in a temperature-controlled environment) and over a much longer period of time (approximately 19 months). The fill used was taken from one residential development in the east of Ireland; the samples tested by Sutton *et al.* (2013) were from a different house in the same development and had a greater original pyrite content. In addition, a database of

chemical test results particular to the same development is used to investigate if the Oxidisable Sulphur (OS) present as sulphides is substantially derived from the pyrite reaction. The rate of pyrite oxidation has been estimated based upon its consumption between the construction date of each dwelling and the date of testing. This investigation considers whether reasonable predictions of the amount of heave experienced can be made from a stoichiometric analysis of the pyrite reaction's chemical equations, in conjunction with (i) a knowledge of the original pyrite content and (ii) the rate of pyrite oxidation deduced from the database.

Chemical test database for development in the east of Ireland

Evidence of gypsum formation

Reid *et al.* (2005) have identified six chemical equations which represent the conversion of pyrite present in parent rock into gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and other reaction products, reproduced in equations (1a-1f). When interpreting chemical test results on pyritiferous fill material, it has been assumed in this paper that the majority of OS present at the time of fill sampling has originated from pyrite without any other significant source of sulphur present. The relevant equations 1a to 1f are given below.



A database (compiled by Aidan O'Connell and Associates Ltd.) comprising geological and chemical test results for 60 dwellings in a development in the east of Ireland is considered in this paper. The properties concerned have experienced severe heave of their floor slabs generating significant structural damage to each dwelling. The geological classification and testing (petrographic description, chemical analysis for total sulphur, water soluble sulphate and acid soluble sulphate) was performed on small samples (10kg-20kg) of the fill which were removed from beneath the floor slab prior to the remediation of the dwelling, the results of which are documented in various reports by Ground Investigations Ireland between 2009 and 2013. The laboratory expansion tests subsequently reported relate to material from one particular property in the same development which was constructed towards the end of 2004, subsequently referred to as Property X.

In equation (2), the relationship between OS, total sulphur (TS) and acid soluble sulphate (ASS SO₄) is provided as set out by Reid *et al.* (2005).

$$OS = TS - (ASS\ SO_4)/3 \quad (2)$$

In Figure 2a, the OS concentration in the fill, calculated using equation (2), is plotted against TS. The strong linear correlation ($R^2 = 0.89$) suggests that the amount of gypsum precipitated is directly controlled by the amount of pyrite present. Although it is possible that there may be small sulphur amounts in organics and that there may have been some gypsum when the fill was placed, these are probably not significant amounts. In this case, the original pyrite is calculated from Equation (3):

$$\text{Original Pyrite} = TS \times 1.87 \quad (3)$$

if no other forms of sulphur are assumed present. If there was no acid soluble sulphur in the rock, the slope of the line in Figure 2a would be unity. However the slope is less (0.588) as ASS rises in proportion to TS as shown in Figure 2b. This relationship spans a wide spread

(0-5%) of original pyrite contents. The data points for Property X are identified in Figures 2a and 2b.

Pyrite consumption rates

As previously mentioned, the demand for crushed stone was very high during the “Celtic Tiger” years; ultimately this resulted in stone being blasted from the quarry face and processed to crushed stone and delivered to site as quickly as possible. In essence this resulted in a very short period of retention of the processed stone within the quarry before delivery to site. Little pre-delivery oxidation of the stone can therefore be assumed.

Accordingly if the original pyrite concentration of the fill (equation 3) was assumed to be intact at the time of placement within a property, the amount of pyrite oxidised between construction and removal of fill at the remediation stage can be quantified according to equation (4):

$$\text{Oxidised pyrite} = \text{ASS SO}_4 \times (1.87/3) \quad (4)$$

In this equation, it is assumed that all liberated sulphur is captured by gypsum with no loss of S or subsequent dissolution of gypsum or other reaction products in the process. In situations where some of the liberated sulphur is lost to this reaction, (this could occur where elevated water levels are present), the effect would be to result in a lower rate of oxidation for identical samples of infill material.

The time between construction and the testing at the remediation stage is subsequently referred to as the period of oxidation. The construction date is assumed as four months prior to the issue of the Certificate of Approval (CoA), for which dates are available. The error associated with this is believed to be 1-3 months, which is small in relation to the period of oxidation, which exceeds 5.5 years in all cases in the database.

This oxidised pyrite concentration, expressed as a percentage of the original pyrite, is plotted against the duration of oxidation in Figure 3a. For 70% of the datapoints, the percentage of

pyrite oxidised lies between 30% and 50%. The same data are reformatted in Figure 3b, where the rate of oxidation is determined as the quotient of the oxidised pyrite concentration and the duration of oxidation. This figure indicates that the fill in 83% of these properties is characterised by oxidation rates in the range 4%/year - 8%/year. Variables such as mudstone lithology, depth of fill, degree of compaction, moisture content and whether or not the house was ever occupied may have contributed to this scatter. The average rate of oxidation for Property X, indicated on Figures 3a and 3b is approximately 6.7%/year.

Expansion experiments

Classification of fill

The pyritiferous fill used in the experiments was sourced from Property X in December 2011 (approximately 6.3 years after construction) when the house was undergoing remediation works for damage caused due to heave of its floor slab.

Geological testing was carried out on a sample of fill taken from Property X, prior to remediation, in March 2011. The tests established the original pyrite content as 2.4% based on equation (3); further test results are provided in Table 1. A description of the main rock types was given for each of the two test samples as part of the geological report. In the samples, there were three different lithologies present. They were:

Calcareous mudstone: Medium to dark brownish grey, commonly flaky and laminated. They are mostly of low porosity but are occasionally porous with abundant openings along grain boundaries. Framboidal pyrite is abundant and some of these frambooids are surrounded by reddish brown alteration rims.

Argillaceous limestone: Predominantly blocky, medium to dark brownish grey, laminated and non-laminated. Many particles contain framboidal pyrite and pyrite cubes.

Limestone: Dense, medium grey particles with low porosity that contain carbonaceous matter and some particles contain crystals/grains of pyrite.

X-ray diffraction testing was not carried out on the sample; however, the report stated that the surfaces of the fill particles were covered in abundant calcareous and argillaceous dust that contained altered framboidal pyrite. Gypsum crystals were present on the surface of some particles of fill. None of the non-acid soluble sulphur was in the form of barite.

A particle size distribution (PSD) was carried out on the fill and Figure 4 shows that the grading for two samples of pyritiferous fill largely fall outside National Roads Authority (NRA) Clause 804 bounds, samples B(i) & B(ii). Also included on Figure 4 is a PSD for the Clause 804 material used in reference tests (sample E).

Experimental apparatus

The fill was delivered to the laboratory at NUI Galway in 500kg synthetic bags and the tests began in January 2012. Six test specimens were set up using the apparatus developed by Sutton *et al.* (2013); a photograph of four of the tests is provided in Figure 5. The experimental setup consisted of HDPE pipes (229mm in diameter) each placed vertically in the centre of a 45L (570mm x 390mm x 280mm) plastic storage box. Water was filled to height, D, and maintained at this level throughout the testing period. The pipes were filled, to a sample height H, above the water level, with compacted fill. The fill was compacted manually to the specified density in 100mm layers by dropping a 7kg tamper evenly across the fill surface. The tamper was dropped from a maximum height of 300mm above the fill surface to ensure that a minimum of fill particles were broken during compaction. The fill was topped with 50mm of compacted sand which supported a 3mm thick by 200mm diameter steel plate. No additional dead or live loading was applied. An independently-supported dial gauge resting on the steel plate measured the expansion/contraction (h) of the fill. The pipes incorporated two rows of 10mm diameter holes drilled 60mm and 100mm from the top of the sand to give the fill access to air with another two rows drilled 25mm and 10mm from the bottom of the pipe to give the fill access to water.

The six tests specimens, all having H=500mm, were prepared to three different target fill densities (nominally 2200 kg/m³, 2000 kg/m³ and 1800 kg/m³) and two different water heights (D=30mm and D=10mm). Relevant details of these tests B1-B6 are provided in Table

1. The results from Sutton *et al.* (2013), labelled A1-A10, are also included in Table 1 for later reference.

The tests were housed in a temperature-controlled room (with an operational range of 10°C-30°C) for the entire duration of the testing, with nominal temperatures in the range 10°C - 20°C applied for tests B1-B6. By way of context, the absolute minimum and maximum mean monthly soil temperatures in the Dublin area, provided by the Irish meteorological service Met Éireann, were 4.6°C and 16.5°C respectively over a 30 year period to 2010, with a mean annual temperature of 10.0°C.

For the first 16 days, a period allotted for self-weight settlement to occur, the room (air) temperature remained at ambient values in the range 17°C-20°C. Between day 16 and day 573, the temperature was held nominally within 1°C within 10°C, 15°C or 20°C in no particular pattern, as indicated in Table 2. In general actual room temperatures remained within 1°C of these nominal values. The hold period corresponding to each nominal temperature value is provided in Table 2. In general, the temperature of the water in the basins was up to 1.5°C lower than the actual room temperature and it typically took 1-2 days for a steady water temperature to re-establish after a temperature change. Readings of the dial gauges and measurements of the water temperature and pH were made at intervals not exceeding 3 days, but much more frequently in some cases in the immediate aftermath of a temperature change. There were two periods without data retrieval, as noted in Table 2. The relative humidity captured from the Building Management System ranged between 54% and 87% with an average of 74%.

Two additional reference tests (E1 and E2) having the same apparatus as B1-B6 with H=500mm and D=30mm were situated in a separate temperature-controlled room (operational range of 10°C - 40°C), see Table 1. The fill used was good quality pyrite-free Clause 804 sourced from a quarry in the west of Ireland and compacted in both pipes to a nominal density of 2000 kg/m³. The purpose of tests E1 and E2 was to help understand the thermal expansion of the entire system (i.e. the fill plus the reference frame) in response to a temperature change.

Results and discussion

Normalised expansion (h/H) is plotted against time for tests B1-B6 in Figure 6 and control tests E1-E2 in Figure 7 with the (room) temperature hold periods also identified. Evaporation-inducing conditions in the temperature-controlled room meant that water levels needed occasional replenishment. This replenishment will have masked the steady reduction in pH levels with time, due to acid production in the reaction process, as displayed by Sutton *et al.* (2013). It is worth noting that the dissolution of atmospheric CO₂ may also have affected the pH levels. However, given that the pH of the added water was nominally 7.2, it would be anticipated that the pH values measured would fall slightly below the range of 8.0 to 8.4 measured by Sutton *et al.* (2013) up to approximately 145 days; measured pH values in this series fell between 7.7 and 8.1.

Effect of density

Since tests A1-A10 (Sutton *et al.* 2013) intentionally targeted a constant density of 2000 ± 50 kg/m³, it was not possible to assess the influence of density on expansion magnitude or rate in that test programme. However, a clear relationship between the overall amount of expansion and density is evident for tests B1-B6 in Figure 6. Over the 573 day period, tests B1 and B2 (nominally 2200 kg/m³) showed the greatest amount of expansion, followed by tests B3 and B4 (nominally 2000 kg/m³). Test B5 (nominally 1800 kg/m³) expanded by a relatively small amount, characterised by long periods without expansion interspersed with short periods of aggressive expansion. Czerewko & Cripps (2006) suggest that gypsum crystals “*grow in areas of least stress such as voids*”, and therefore it is conceivable that as the pyrite reacted to form gypsum, the crystals precipitated in the more ample void spaces available in the lower density material, which did not cause a sufficient build-up of pressure to manifest in measurable heave until much later than for the higher density specimens. The dial gauge on test B6 (also nominally 1800 kg/m³) only operated satisfactorily after day 304, and the subsequent movement registered has been relatively smooth since day 351.

The effect of density can be established more clearly by plotting average expansion rates corresponding to periods at constant temperature against density in Figures 8a (nominally

10°C), 8b (nominally 15°C) and 8c (nominally 20°C). These average expansion rates exclude the first week data at each temperature (to exclude effects of thermal expansion/contraction discussed later). While it is acknowledged that these simple average rates are computed for widely different time periods (ranging from 27-7=20 days to 223-7=216 days), the strong influence of density on expansion is evident. This is consistent with empirical field observations by Aidan O'Connell and Associates for houses currently being repaired; infill with low levels of compaction appear to be suffering lower levels of damage versus those with higher levels of compaction for infill with equivalent levels of pyrite/TS. It is also acknowledged in Tuohy *et al.* (2012) that the degree of compaction had an effect on the extent of damage by “*providing no leeway for expansion*”.

Effect of temperature

A close examination of the data in Figure 6 reveals a subtle immediate expansion or contraction response to many of the instances of temperature change which are transient in nature. A transient response to temperature changes is also observed in Figure 7 for the pyrite-free reference specimen. In order to examine the short term response of the tests in greater detail, the displacements during the days immediately after a temperature change are plotted in Figures 9a (B3-B4) and 9b (E1-E2); a temperature drop from 20°C to 15°C is used as a representative example.

In both test sets, a sudden increase in movement is observed followed by a gradual decrease of similar magnitude over approximately 30 hours. The former is believed to be due to the immediate thermal contraction of the aluminium upright of the dial gauge support frame, and can be predicted accurately using the coefficient of thermal expansion for aluminium (22.2×10^{-6} m/m °C). This downward movement of the dial gauge frame gives the impression of “apparent fill expansion”. The latter is believed to be due to contraction of the fill material which equilibrates more slowly than aluminium to the new air temperature. While the magnitudes of expansion in Figures 8a and 8b are similar, they should not be compared directly as the interval to the first measurement after temperature change is not the same in both cases (1.5 hours for B3, B4; 15 mins. for E1, E2). It should be noted that for tests B5

and B6, the upright of the dial gauge frame is made from steel having a coefficient of thermal expansion of 13×10^{-6} m/m °C and the movement patterns differ accordingly.

To consider the nature of the expansion systematically, average expansion rates were computed over uniform consecutive intervals of one week from the start of each change in temperature. In light of the thermal effect identified above, average expansion rates from the first week after a temperature change (time $t \leq 1$ week) were excluded. As a result, the 9-day hold-period 1 at 15°C was deemed too short to contribute useful data.

In Figure 10, the weekly displacement rates (quoted in mm/day) for $t > 1$ week are plotted against the average water temperature over the week for tests B1 to B6. The data exhibits some scatter as would be expected over a 573 day testing period, but for these data, an effect of temperature is not apparent, especially for the datasets at nominal temperatures of 10°C and 15°C. It should be noted that while the rates seem to be marginally lower at the 20°C temperature, there are fewer data points for fair comparison, and the stage of the experiment may also be relevant. This important insight into temperature effects was not possible from the Sutton et al (2013) series where the temperature was constantly changing.

In Figures 11 (a-d), the data in Figure 9 are reproduced; this time grouped by the stage of the experiment (1-200 days, 201-400 days and 401+ days) to assess whether the rate of reaction has changed with time. Data points for days 1-200 only include rates at 10°C and one at 20°C (the 15°C period was not of sufficient length as mentioned). Data points for days 201-400 only include periods at 10°C and 15°C but data for days 401+ includes periods at all nominal temperatures. These figures suggest there may be a reduction in reaction rate after 400 days, i.e. 8.5 years after the fill was used in construction, possibly due to the depletion of pyrite available for oxidation. However, caution is advised given the variable number of data points for each temperature/time combination. By way of comparison, Maher *et al.* (2011) reported that rates of reaction had not abated in their experiment after a two year period.

Effect of water depth

In Figure 6, it is clear that the specimens having D=10mm submerged depth (i.e. B1 and B3) showed greater expansion than those with D=30mm submerged depth (i.e. B2 and B4). In

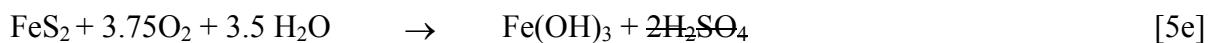
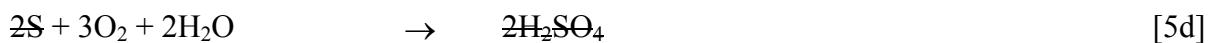
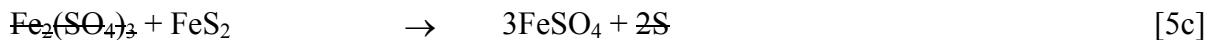
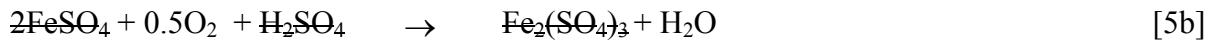
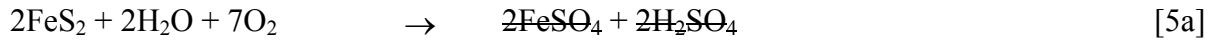
Figure 12, submerged depth D is plotted against normalised expansion rate h/H for tests B1-B5 and these data are supplemented by tests A1-A10 (Sutton *et al.* 2013). For some measure of temperature compatibility, the data used for the B tests is taken from period 2 at 10°C, while a 6.5 week period when the average temperature was 10°C (min. 9.1°C, max. 11.9°C) was extracted from Sutton *et al.* (2013) data. The combined dataset suggests that sensitivity to water depth in this experimental setup only arises for D<30mm, with greater expansion occurring with reduced depth. This is in keeping with the observations of Hammock and Watzlaf (1990) who stated that saturating pyritic material could reduce oxidation rates by 96%. It is possible that the moisture already in the fill at the time of construction and the subsequent moisture in the air are sufficient for the oxidation process to take place.

A theoretical model of pyrite consumption and resulting expansion

In this section, the expansion rates measured in the experiments on the fill material are benchmarked against a theoretical model of the chemical conversion process involved. The model considers the increase in the height of the sample as a result of the chemical conversion only, referred to subsequently as the “equivalent heave height”, and does not consider the potential additional volume increase that arises from the opening of naturally-occurring laminations through the forceful growth of gypsum which only partially fills these voids. It is assumed that the chemical conversion occurs in the same unit of fill, although in reality, this is unlikely as oxidation and precipitation require different environments.

The model is based upon a molecular/molar analysis of equations (1a-1f) from Reid *et al.* (2005). Equation (5) is an adaptation of Equation (1) with multipliers to (1a), (1d) and (1e) and “strikethroughs” to identify a reaction product used up in another reaction. The remaining reaction products are listed in Table 3 along with their molar ratios which are relevant to height change calculations. The equivalent heaved height is assumed to derive from the height change induced by reaction products $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, Fe(OH)_3 and FeSO_4 less than due to the original FeS_2 and CaCO_3 . Mineral molecular masses and densities relevant to the calculations are also shown in Table 3. The full set of calculations is set out in Table 4; these are based on the pipe dimensions outlined previously and four different sample densities are

chosen to correspond to those of laboratory tests B1-B4. An original pyrite content of 2.36% for Property X (Table 1) deduced from the TS value of the fill tested is assumed. The net model expansion is converted to a theoretical rate of expansion based upon the deduced average pyrite oxidation rate of 6.7% per year (Figure 3b) for Property X.



The theoretical rates of expansion in Table 4 are compared in Figure 13 with those measured in the laboratory tests that were presented in Figure 9. These results summarised in Table 5. In the interests of consistency only data for one temperature (nominally 10°C, which is the mean annual soil temperature as mentioned in Section 3.1) is used and the data plotted represents an average of those in Figures 10a and 10b.

Sample B4 has the lowest density and its expansion matches the theoretical model most closely. As the density increases, the difference between the theoretical and measured expansion rates dramatically increases. The results strongly indicate that gypsum crystal growth will occur in freely available void space between the stone particles initially and additional void space is generated within the laminations of the rock material and is generated more quickly as the density of the sample increases. This matches what has been observed in houses currently under repair; where the infill appears to be loosely compacted, the onset of damage is delayed compared to a property where the infill is well compacted, with equivalent levels of pyrite/TS.

From Figure 13, it can be seen that the average expansion corresponding to a density in the middle of the range tested (i.e. 2100 kg/m³) is 1.55 mm/year. It should also be noted that the

measurements presented in Figure 13 are likely to form upper-bounds to those likely in practice. In the laboratory experiment, the friction between the fill and the pipe will be relatively low and no additional live or dead load has been imposed on the sample throughout the period of observation. Another interpretation of this figure is that additional empty void space is generated through the precipitation of the gypsum crystals.

Conclusions

In this paper, the research of Sutton *et al.* (2013) in a novel apparatus designed to model fill in an underfloor environment has been extended to consider the effects of temperature, density and water height more systematically. New findings include:

- (i) At constant temperature, the density of the fill appears to have a significant impact upon the onset time and/or magnitude of pyritic heave occurring.
- (ii) While changes in temperature appear to induce thermal expansion/contraction in specimens, the long term expansion rates appear to be largely independent of temperature for the range considered.
- (iii) It appears that a lower submerged depth of sample, based upon a combined review of the Sutton *et al.* (2013) tests and the new tests presented in this paper, can actually lead to a greater rate of reaction even though water (in vapour or liquid form) is required for, and consumed in, the reactions.
- (iv) There is evidence that the rate of reaction may be slowing beyond 400 days since the beginning of the laboratory experiment, i.e. almost 8.5 years after the fill was used in construction.

In addition, a review of chemical test results for 60 properties on the development from where the tested pyrite was sourced is used to confirm that the pyrite content of the rock controls the amount of gypsum produced and in turn the extent of expansion. A molecular/molar analysis of the pyrite chemical process equations, in addition to a knowledge of the original pyrite content and rate of oxidation, has been used to give a frame of reference for the amount of heave experienced in the laboratory experiments. It is concluded that, since

the net increase in volume due to precipitation of products is significantly less than the actual expansion, precipitation of products creates porosity.

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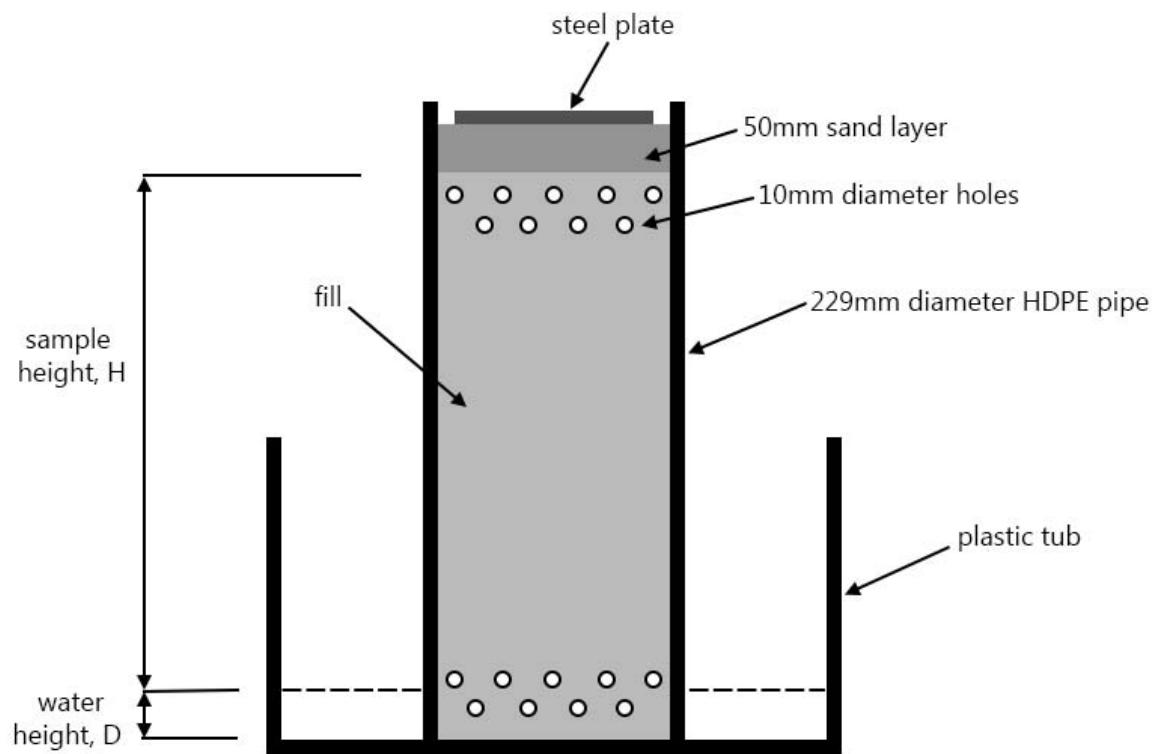
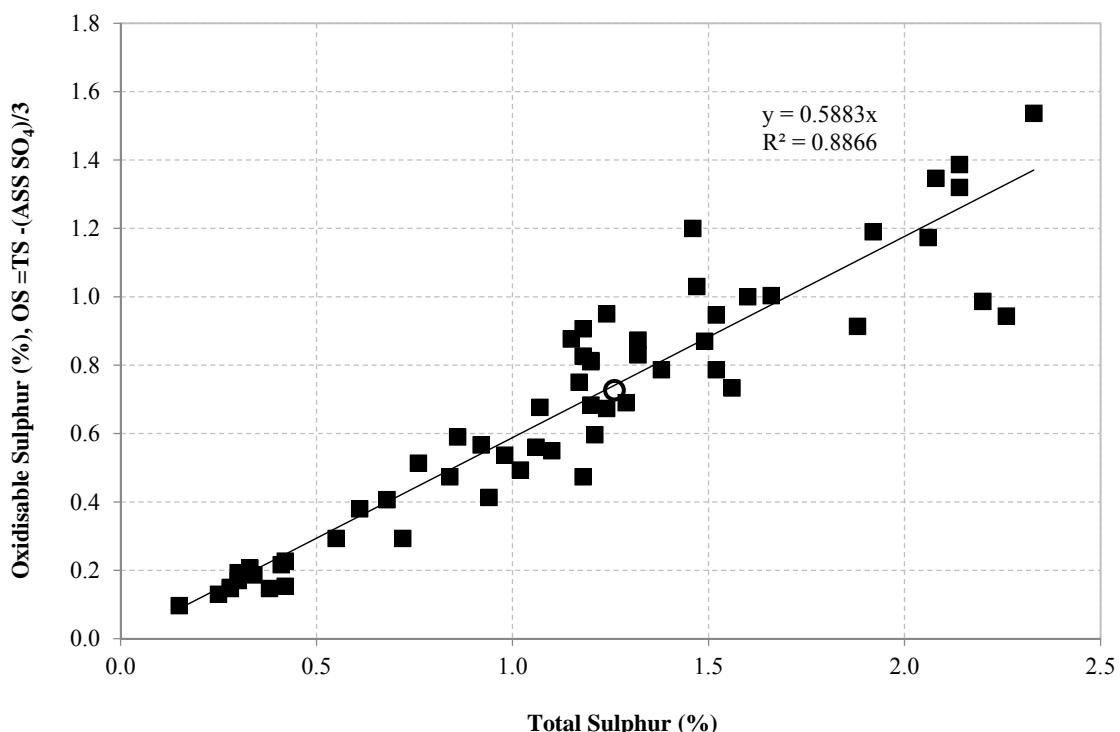
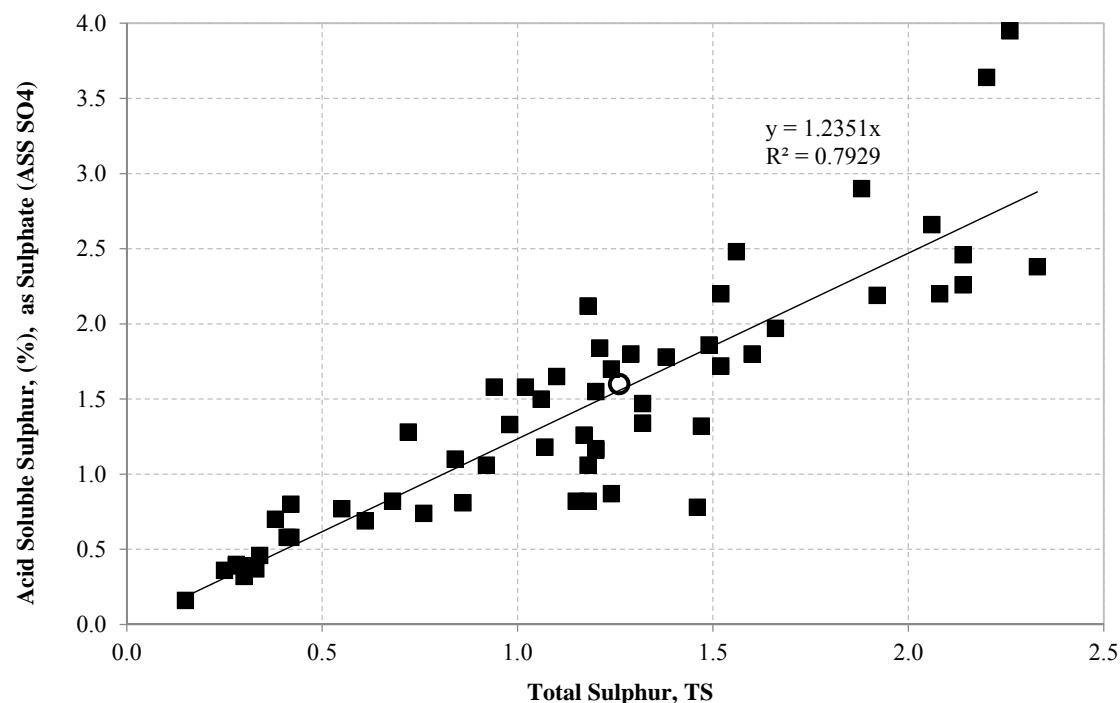


Figure 1: Schematic of the testing apparatus used

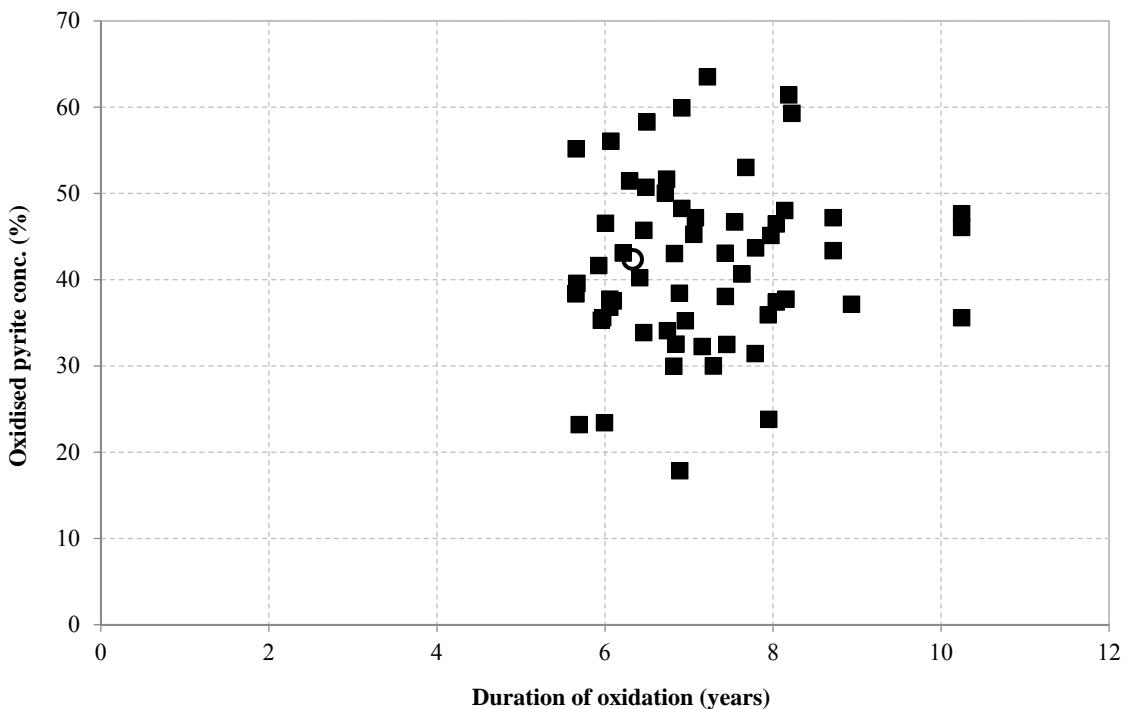


a)

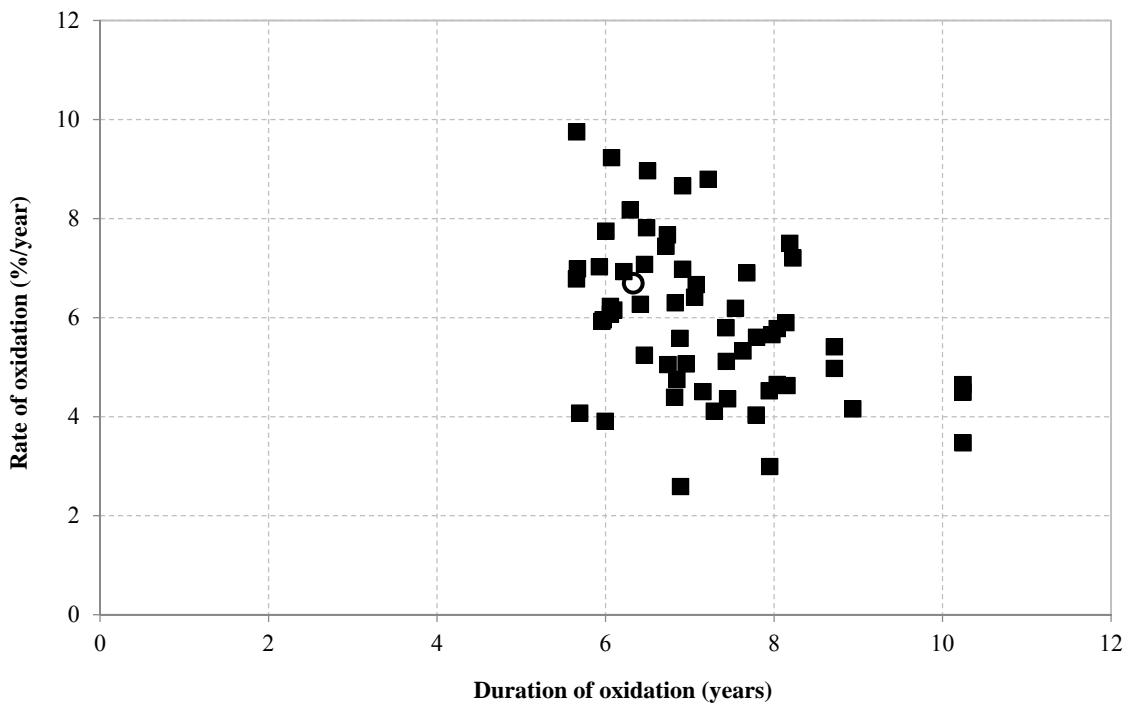


b)

Figure 2: Plot of chemical test results (all from the same development and carried out by the same laboratory): a) Oxidisable sulphur v total sulphur; b) Acid soluble sulphur, as sulphate (ASS SO₄) v total sulphur



a)



b)

Figure 3: a) Oxidised pyrite concentration v duration of oxidation; b) Rate of oxidisation v duration of oxidation.

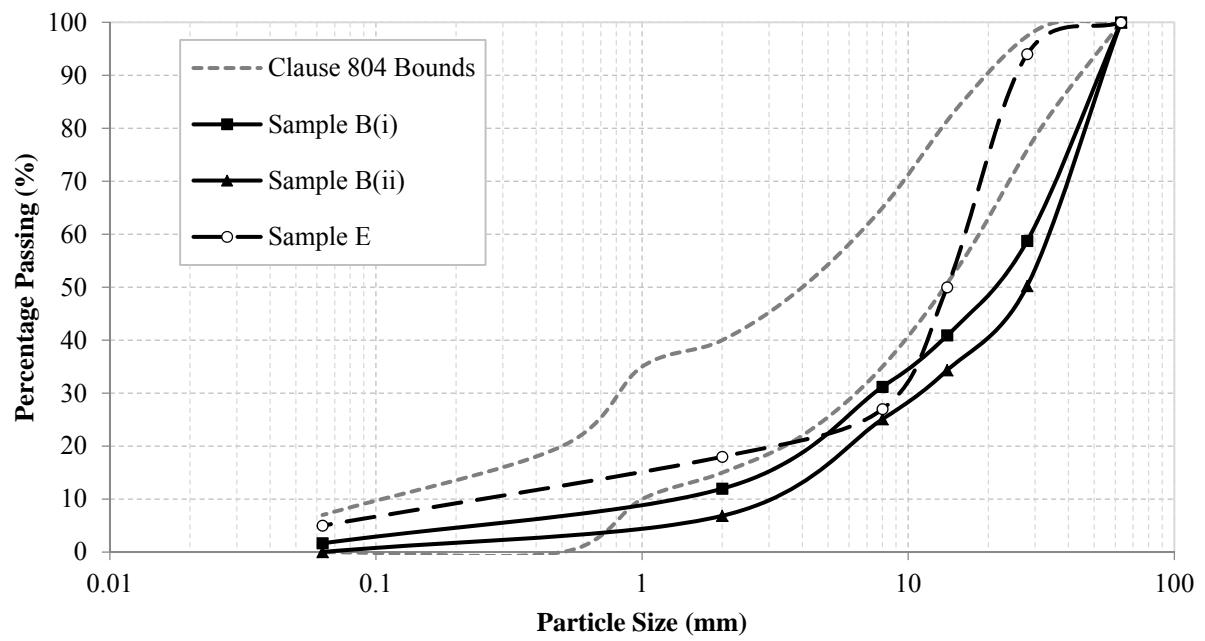


Figure 4: Particle size distribution of the fill used compared to Clause 804 specifications

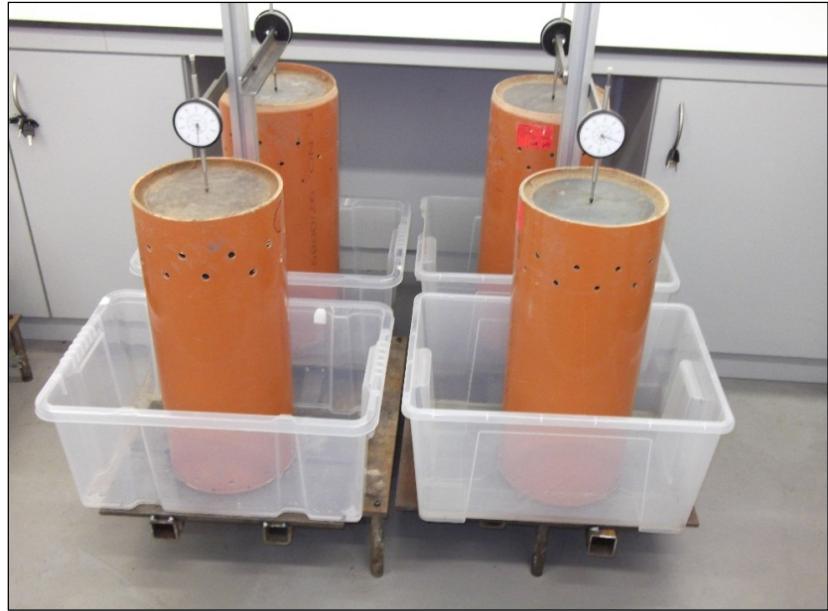


Figure 5: Photograph of tests B1-B4

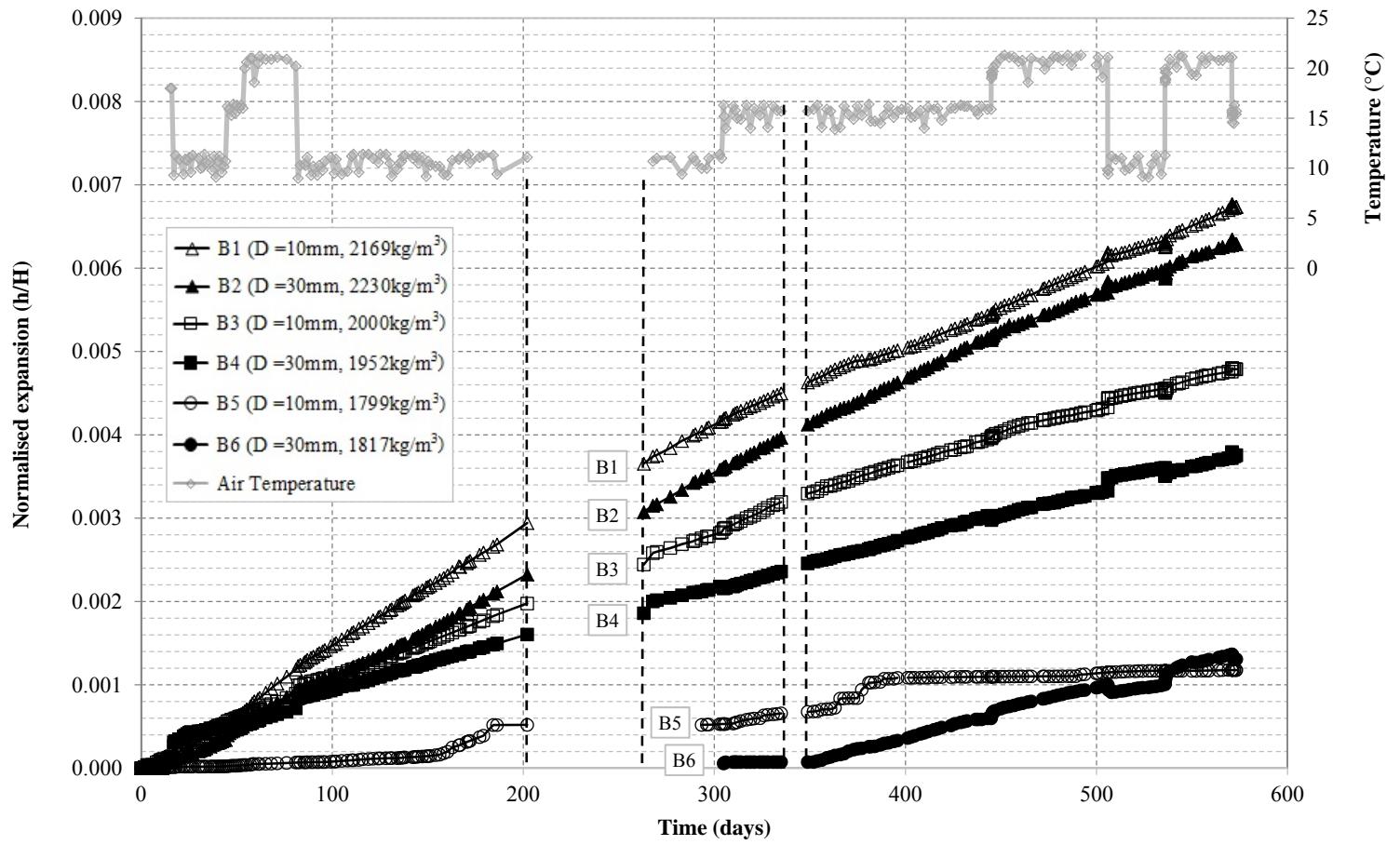


Figure 6: Normalised expansion (h/H) and room temperature versus time (B1-B6).

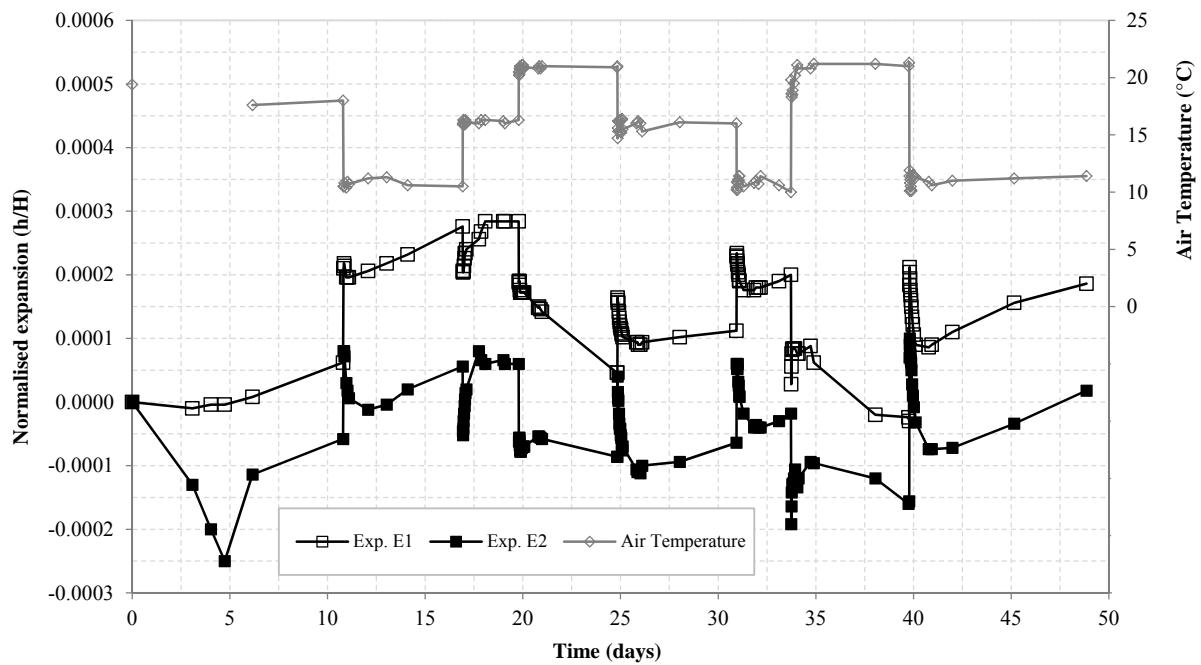


Figure 7: Normalised expansion (h/H) and room temperature versus time for pyrite-free tests (E1-E2).

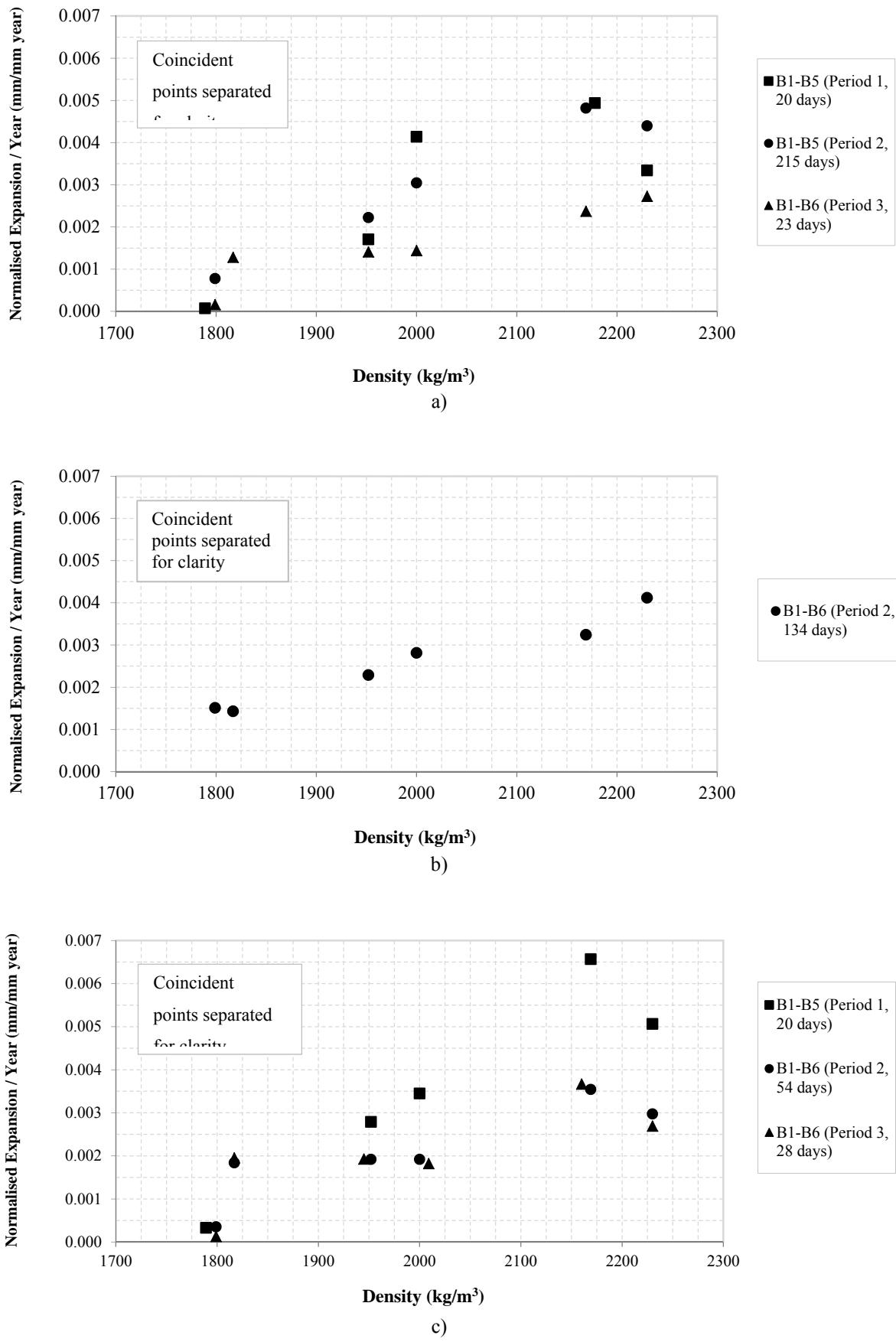
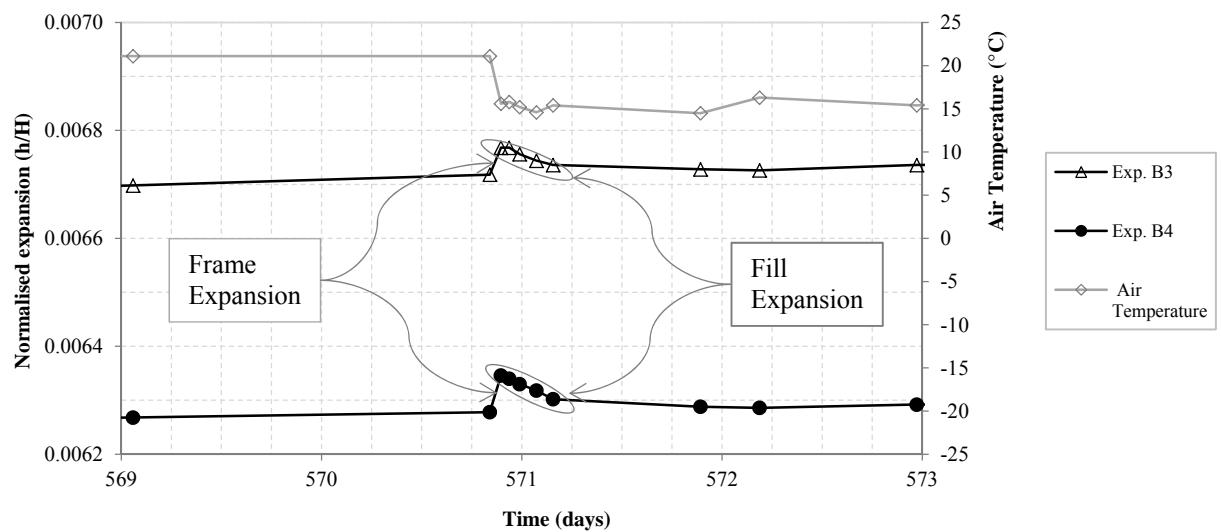
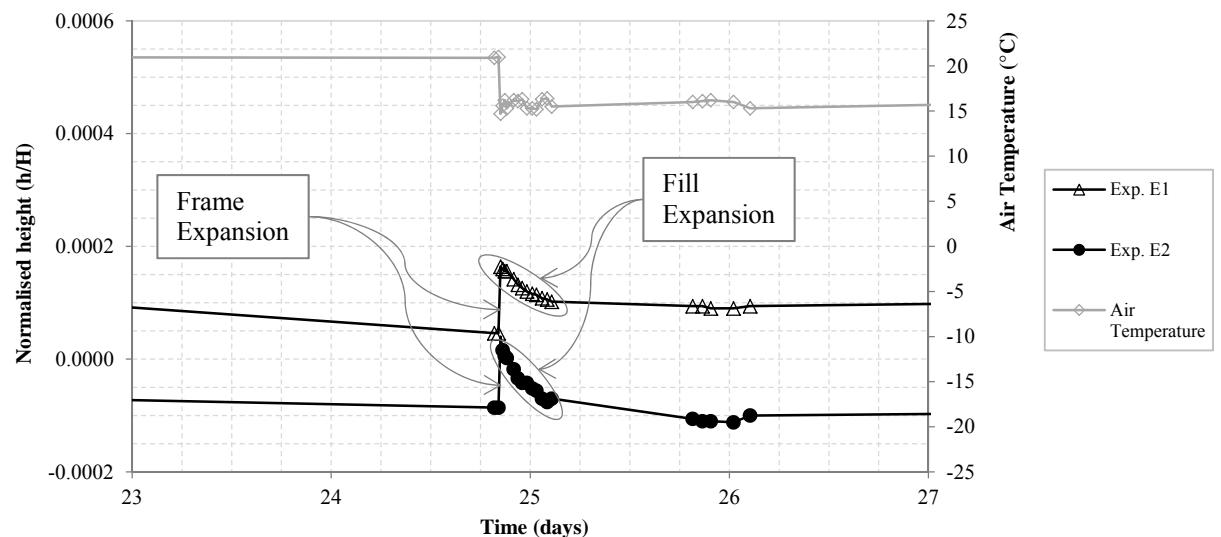


Figure 8: Normalised expansion, excluding expansion that occurred during the initial week, for a) periods at 10°C; b) periods at 15°C; c) periods at 20°C.

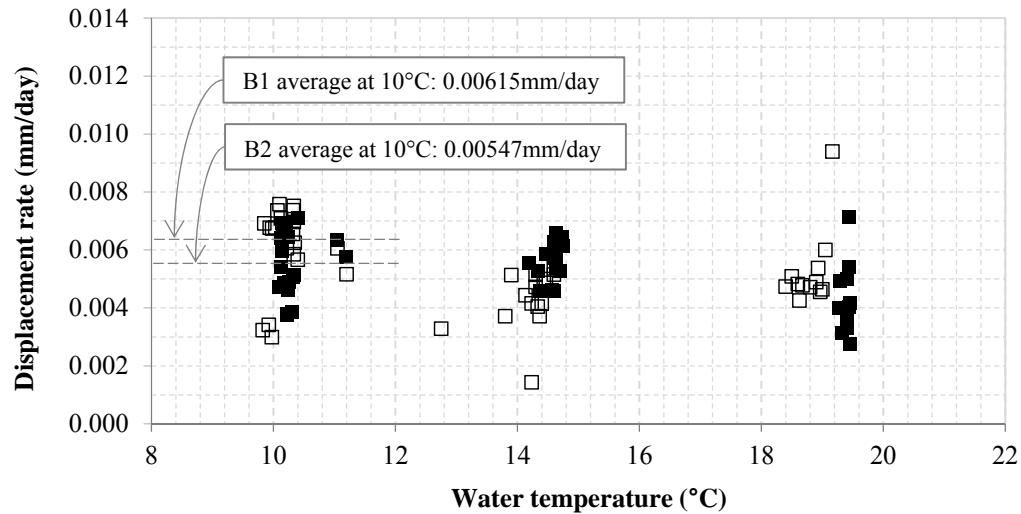


a)

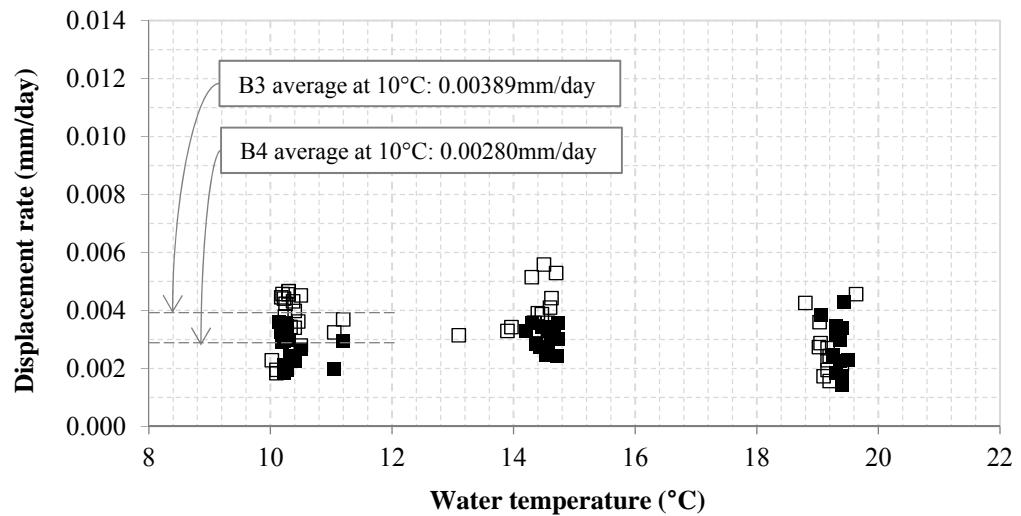


b)

Figure 9: Normalised displacement after a temperature change from 20°C to 15°C in tests a) B1-B4 and b) E1-E2.



a)



b)

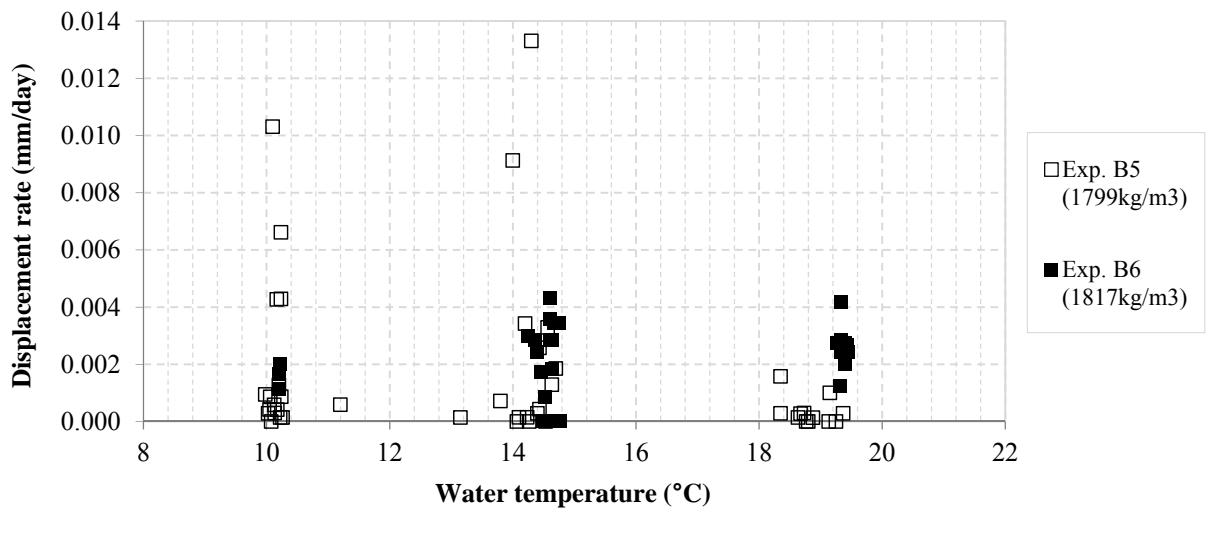
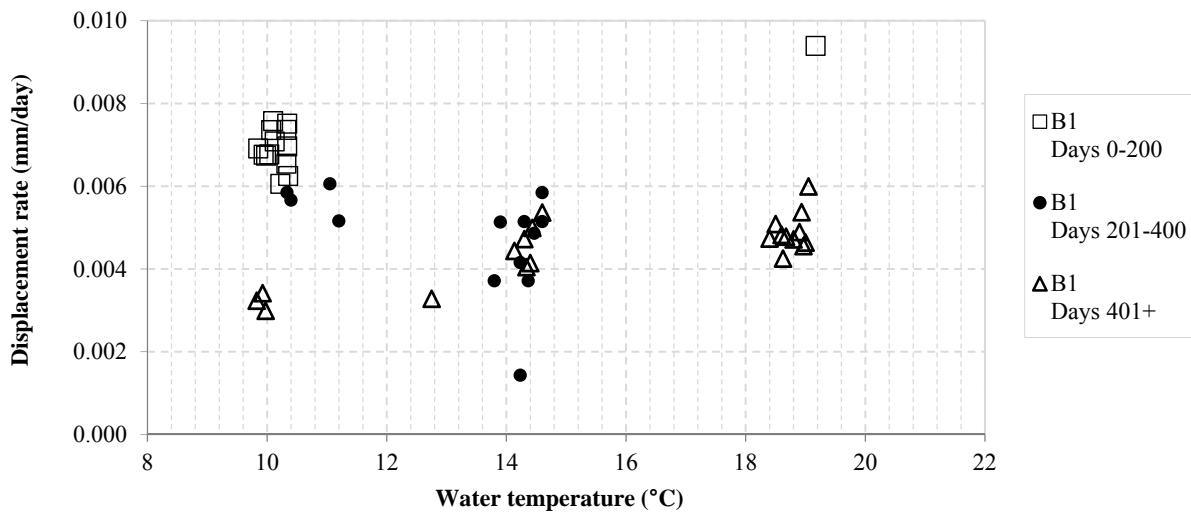
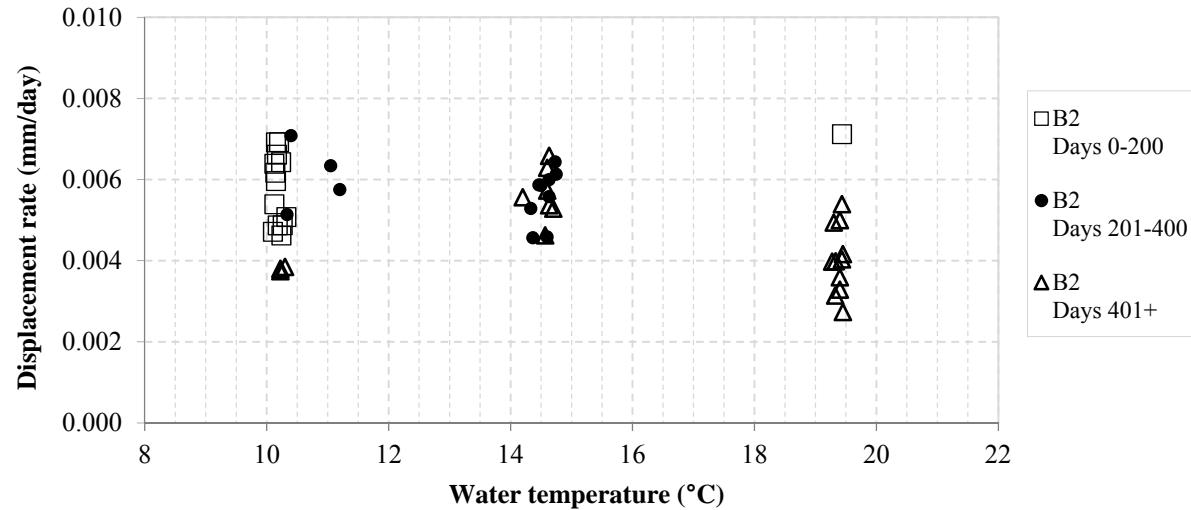


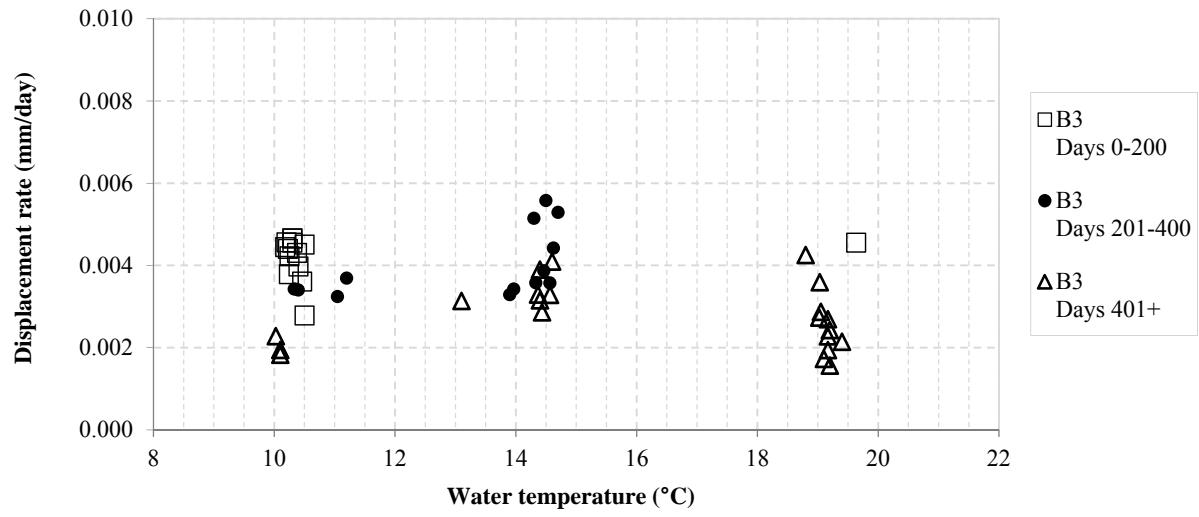
Figure 10: Normalised expansion, excluding expansion that occurred during the initial week periods, for tests a) B1-B2; b) B3-B4; c) B5-B6



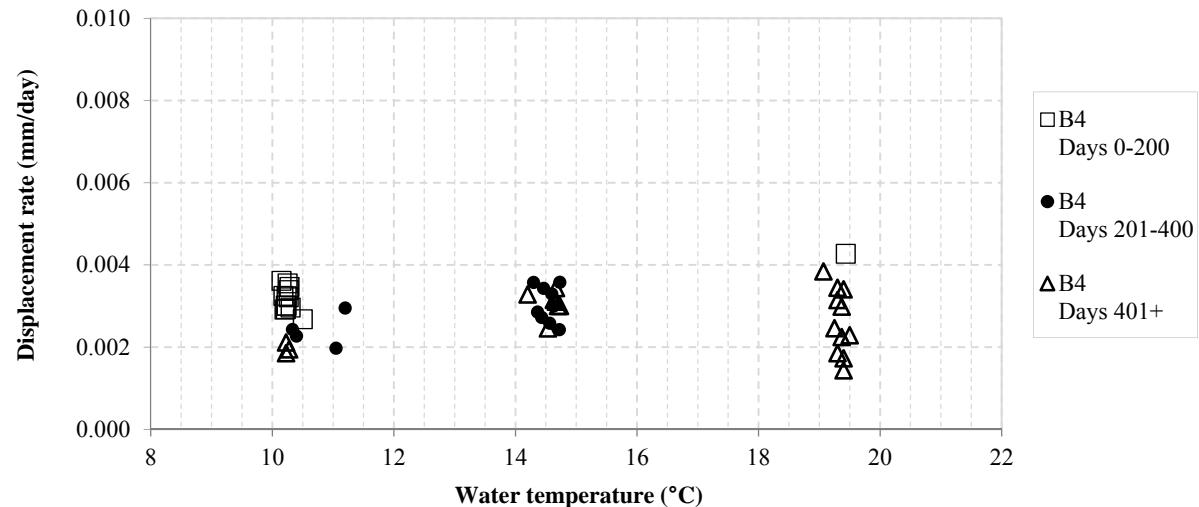
a)



b)



c)



d)

Figure 11: Weekly displacement rates subdivided into days 0-200, 201-400 & 401+ for a) B1; b) B2; c) B3 and d) B4.

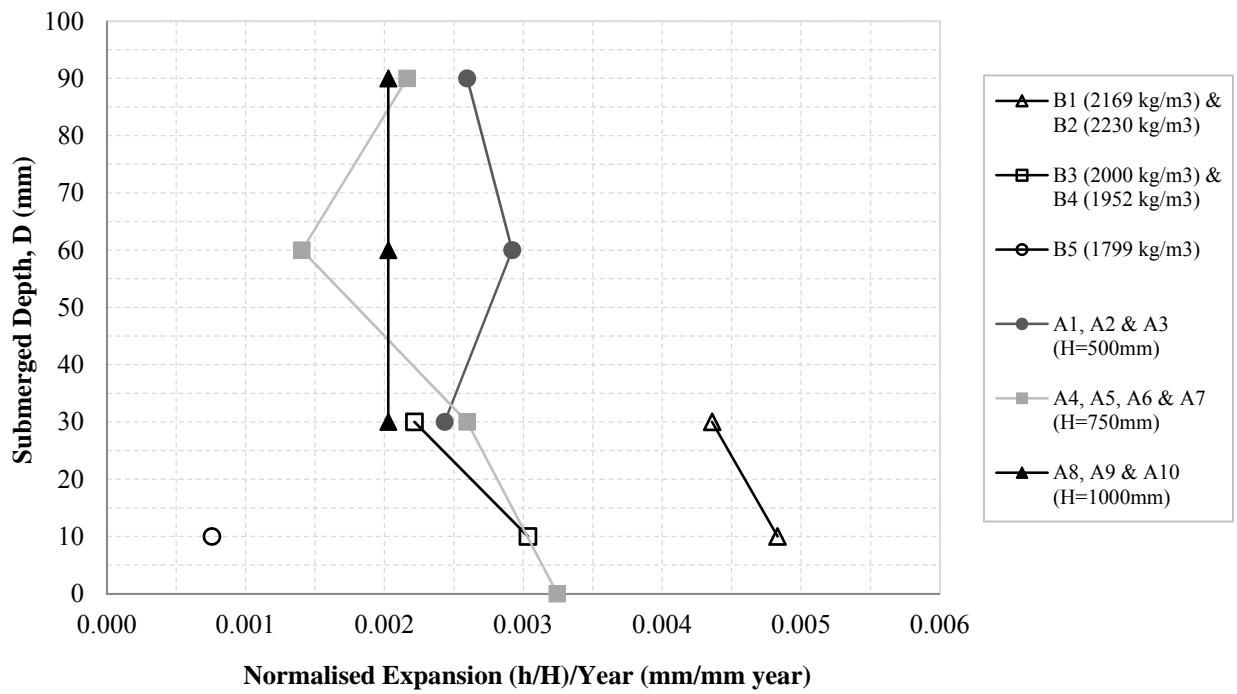


Figure 12: Normalised expansion versus water height for period 2 at 10°C and a 6 ½ week period from Sutton *et al.*, (2013).

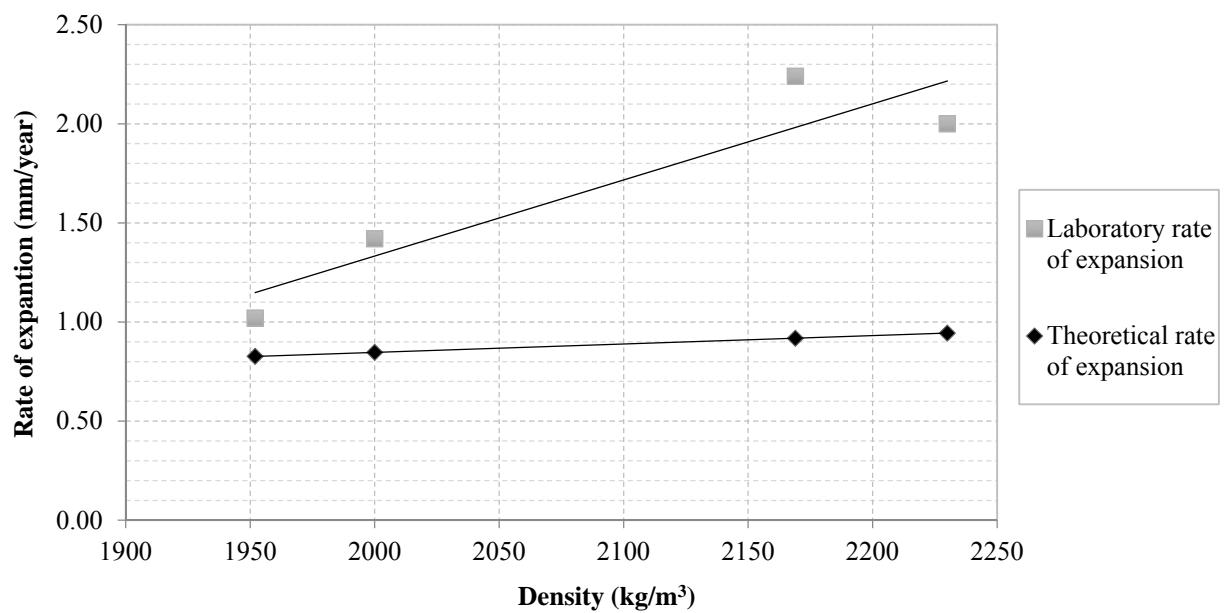


Figure 13: Laboratory and theoretical rates of expansion versus density