Revitalising Collyweston limestone slate production by artificial freeze/thaw splitting

LAYCOCK, Elizabeth <http://orcid.org/0000-0003-3758-6829>, JEFFERSON, David, HETHERINGTON, Steven <http://orcid.org/0000-0002-9851-8478>, CLEGG, Francis <http://orcid.org/0000-0002-9566-5739> and WOOD, Christopher

Citation:

LAYCOCK, Elizabeth, JEFFERSON, David, HETHERINGTON, Steven, CLEGG, Francis and WOOD, Christopher (2017). Revitalising Collyweston limestone slate production by artificial freeze/thaw splitting. Construction and Building Materials, 159, 486-498. [Article]

Revitalising Collyweston limestone slate production by artificial freeze/thaw splitting

AUTHOR

Laycock, Elizabeth Anne [a]; Jefferson, David [b]; Hetherington, Steven [a]; Clegg, Francis [a] & Wood, Christopher [c]

Affiliations

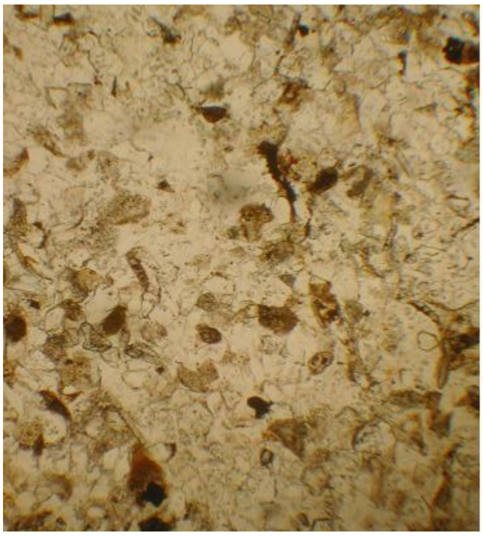
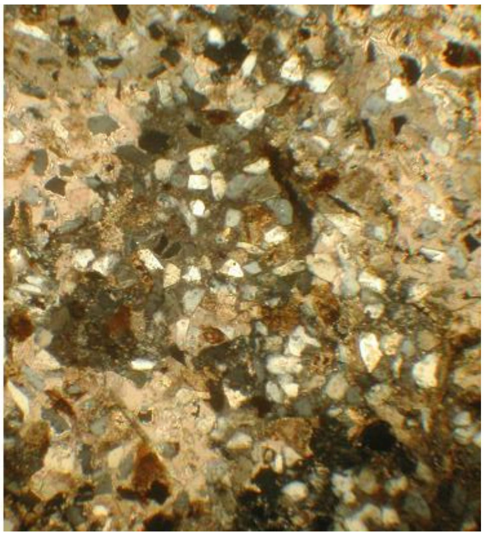
a. Sheffield Hallam University, Howard Street, Sheffield, S1 1WB E.A.Laycock@shu.ac.uk
b. Jefferson Consulting; The Old Armoury, Crown Business Park, Old Dalby, Melton Mowbray, Leicestershire, LE14 3NQ enquiries@Jeffersonconsulting.co.uk
c. Historic England The Engine House, Fire Fly Avenue, Swindon SN2 2EH

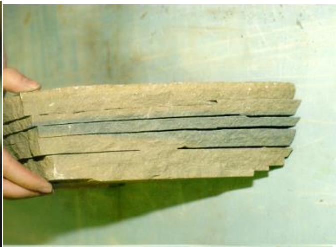
Corresponding Author **Laycock, Elizabeth Anne**
+44114 225 3200
Howard Street, Sheffield, S1 1WB E.A.Laycock@shu.ac.uk

1. Abstract

Extraction of limestone roofing slate from Collyweston was an industry which was considered extinct by the early 1990's, with building repairs relying on wholesale recycling of roofing from demolished buildings. Traditionally stone extracted from the mines was exposed to natural cycles of freeze/thaw to facilitate splitting. Work was undertaken over several years to investigate the resources available and whether these could be artificially frozen to produce slates. The work identified a freeze/thaw regime which could be used to produce roofing slates for historic buildings and which were used in the Historic England restoration of Apethorpe Palace. Mining of the Collyweston limestone stone has now resumed and uses artificial freezing to achieve production of slates which is economically feasible due to the faster process time.

2. Graphical Abstract


3. Highlights

- Historically the extraction of Collyweston limestone roofing 'slates' (tilestones) was important from the Roman period onwards.
- Various factors influence the efficacy of splitting including the block ('log') morphology and size, length of time since extraction and freeze/thaw regime.

- The commercial production of tilestones from the Jurassic Lincolnshire limestone is possible by artificial freeze-thaw processing.

4. Keywords

limestone; roofing; slate; stone; mining; conservation; freeze/thaw; vernacular; Collyweston; tilestone

5. Introduction

Limestone and sandstone roof tiles (tilestones) have been used in Britain since at least the Roman period. The Collyweston limestone, together with the Stonesfield Slate from the Cotswolds, are almost unique in that traditional methods required the stone to be exposed to winter frosts in order to allow the material to be split manually into thicknesses which vary considerably but are estimated at around 15 mm. This paper presents the findings from a series of ongoing investigations, commenced in 1998 and completed in 2014, into the feasibility of using artificial frosting in order to produce tilestones. The conservation of buildings, especially those which are Listed or within Conservation Areas, relies on the adequate supply of appropriate materials and the use of salvaged material is not sustainable in the long term. As part of the work a feasibility study was carried out into the mineral resources available, as well as extensive frost testing both in the laboratory and in pilot plants.

6. Literature

Modern roofing materials are normally thought of as consisting of 'slates', a geological term applied to naturally formed metamorphic rocks, or ceramic tiles which are manufactured. Historically, natural stone materials (other than slate) used for roofing in Britain were sedimentary rocks including sandstone and limestone, which were available in thin sheets and had low porosity (Historic England 2005). The Collyweston limestone (also termed Collyweston slate) is one example within the Jurassic calcareous 'slates', which are calcareous sandstones or sandy limestones. Technically these should be referred to as flagstones or tilestones, and the latter term is adopted in this paper. The climate during the Jurassic period was warmer than at present with thermophillic organisms such as corals and extensive tropical carbonate belts (Selwood & Valdes, 2008). Depositional environments were predominantly either shallow marine or non-marine with open marine sedimentation only persisting in the southern areas of the British Isles. In overall form the surface consisted of a shallow ramp facing to the south west in which a number of basin areas with different sedimentary systems developed in response to sea level change (Hesselbro, 2008). The Inferior Oolite Group formed in the Worcester Basin and on its shallow water margins is the parent unit of the Lincolnshire Limestone (Hesselbro, 2008; BGS 2016). According to King (2010) the Lower Lincolnshire Limestone is a fine grained cream, pale yellow to yellowish-beige, or darker greyish limestone. This unit is of Bajocian age and occurred within the Middle Jurassic period formed between 170.3 ±1.4 Ma and 168.3 ±1.3 Ma (ICS, 2013). Typically the Lincolnshire Limestone is comprised of calcilutites, sandy limestones, peloidal wackestones and packstones in the lower parts of the sequence (BGS, 2016).

The Collyweston limestone is found in the base of the deposit and Historic England (2011) date its use to Roman times since Collyweston roofing has been found at places such as Drayton Roman Villa and the 3rd century basilica at Godmanchester. Archival evidence shows that in 1375 9,500 tilestones from ‘Colyn Weston’ were used at Rockingham Castle and in 1390 a further 4,500 ‘sclastones’ were supplied to the castle. Oakham Castle also purchased 5,000 ‘sklat’ at Collyweston in 1383 (Salzman, 1952). However, by the 1920s there were only six slating pits in operation (Goodwin, 1987) and by 1967 this had been reduced to only three pits (Purcell, 1967). It is estimated that Collyweston tilestones roof at least 1500 buildings in northern Northamptonshire alone, and their use extends into adjoining counties (Historic England, 2011). Until very recently intermittent working only occurred at a single mine. The horizon from which the stone is extracted is normally less than 1 metre thick and underlain by quartz sand. It is known to occur in an area stretching from Burghley House, south-west on the south-east side of the Welland valley to Wakerley, and south east of this line at least as far as Apethorpe. Extraction occurred wherever the stone was suitably fissile and the original stone was worked at outcrops on the side of the Welland valley. However, once the outcrop exposures had been depleted, the strata was followed underground, locally termed 'foxing' (Woodward 1894:182), using drifts, locally termed ‘fox-holes’ (Judd, 1875). The main areas of this development being around Collyweston, Easton, Dene and Kirkby (Woodward 1894:170) and Duddington (*ibid* 483). Extraction was also undertaken by quarrying, but drifts steadily became the predominant method. Unlike the stone at outcrops, that found at depth down drifts showed little or no tendency to split naturally. In order for this limestone to be split into thin sheets suitable for the manufacture of tilestone, it had been discovered that it was necessary to subject the extracted stone to a series of frosts.

Both drifts and shafts extended below the limestone and into the sands of the underlying Grantham Formation. Miners had to lie on their sides, picking away at the relatively firm compacted sand under the limestone bed, using a tool known as a foxing pick. The limestone was supported during the removal of the sand with temporary supports, in the form of columns of waste stones (Historic England, 2011). The presence of a joint set with a strike of 40º West and another less pronounced at right angles allows a section of the limestone to break from the bed when sufficiently undermined (Judd, 1875:182). When the overlying bed could be heard to be 'talking', that is issuing a series of clicks; it would be ready to fall, ideally at the end of a day's foxing. The miner would then retreat removing the temporary supports as he went. The undermined part of the seam would then fall, hopefully breaking into easily managed pieces of stone known as 'log'. If the seam did not fall, steel wedges would be driven into it and a bar known as a 'lion's tail' would be used to lever the seam down. The log was taken by a barrow, known as a shim, to the surface or to the foot of the shaft (Collyweston Stone Slaters' Trust, 2001). Where mining was carried out the galleries were supported by piles of waste stone (Woodward 1894: 197) in the manner of pillar and stall. Long face working was used and these walls ran parallel to the face. As they were constructed, the excavated sand and waste stone was placed behind them providing, not only a roof supporting pack in the worked out area, but also somewhere to place all the waste stone immediately adjacent to the working area, removing the necessity for transporting any waste at all to the surface. All the stone carried out of the mine had therefore been selected as being suitable for the manufacture of roofing material.

It has always been known that it is essential that the log should remain damp so that freeze-thaw cycles could initiate splitting, those which dried out prior to frosting were noted to lose their fissile properties. Log for the tilestones were therefore worked over the winter period (ibid: 194), usually the six to eight weeks in December and January. This extraction period was also limited by the fact that the water in the pits rises in the mines during the spring making working impossible. The logs were laid on the grass and kept watered so that the 'water of the quarry' (also known as 'quarry sap') would not evaporate before frosting. A rapid succession of sharp frosts and thaws was found to be most favourable to tilestone production. Large barrels of water were often located in the frosting area in order to supply water to keep the logs wet. Judd (1875) reported that "three or four good frosts" were needed and "as a rule of thumb, a week's frost is needed." After frosting, tilestones were cleaved and trimmed to the required shape and size using hand tools. Larger tilestones were attached by means of a wooden peg with diminishing courses towards the ridge where the small tilestones could be fixed with mortar (Judd, 1875:182). Since the log varied in size, the resulting tilestones also varied in size.

They were graded, and named, into 27 different sizes, ranging from 6 inch “Even mopes” to 24 inch “Long tens” (Purcell, 1967). This was normal practice when tilestones were being produced since, in order to reduce waste, every riven piece of stone had to be used.

There are 26 named Cotswold Limestone Slates and up to 27 Stonesfield Slates, the latter being similar to Collyweston in that it also has to be frosted (Historic England, 2005; Ashton, 1980). The Collyweston Slate was described as a buff and blue-hearted stone 'so that some of the slates are blue, others yellow, and many are parti-coloured' although the tilestones were 'said to darken on exposure' (Woodward 1894:484). The industrial production of Collyweston tilestones was reported to be in decline by Judd in 1875 owing to competition from Welsh slate and is virtually extinct at the present time. The lack of current extraction of suitable quality stone has led to the use of existing material for the conservation of historic buildings comprising those salvaged and stored from the original building or second hand where provenance is known Cox (2009). Traditionally, the production of Collyweston tilestones was a labour intensive and seasonal activity, resulting in a relatively low yield. Furthermore, it was dependent upon a series of hard frosts during the extraction period. The fact that the success of the process is dependent upon the retention of natural moisture in the stone between its extraction and successful frosting, also added an extra problem to the process. In a modern commercial context, such a process is unsustainable. If the production of traditional Collyweston tilestones is to be restarted in an economically sustainable manner, it will be necessary to produce the product throughout the year. The possibility of artificially frosting Collyweston stone was therefore considered. This was carried out under controlled conditions, using commercial freezers, but since the process is not random, clearly being dependent upon log size, freezing and thawing regimes and possibly other factors, careful control of the humidity and freezing and thawing rates was required to optimise conditions for splitting with least waste material being generated.

7. Materials and Methods

Petrography

Petrographically the two stones which are split using natural frosts, the Collyweston and the Stonesfield are extremely similar. Although the Collyweston stone can be slightly variable in composition, it is composed typically of about 50% quartz clasts set in a crystalline calcite matrix which makes up most of the remaining 50% of the stone. An important feature related to its ability to split is that it is laminated. These laminations when viewed under the microscope are 1 - 2 mm thick and represent individual thin layers of sediment. However, even after frosting, the stone does not necessarily split along every lamination. Many of the laminations appear to have a certain amount of cavitation, contain scattered grains of mica (a fissile laminated phyllosilicate mineral) and may contain a little more iron than normal. These features may represent a slight pause in sedimentation or a period when erosion was greater than sedimentation.

The siliceous nature of the Collyweston stone is due to the fact that it is the basal bed of limestone in the Stamford area. Typically about one metre thick, the horizon represents a transition zone from the underlying Grantham Formation, which consists of sand in this area, and the overlying oöidal limestone which includes such horizons as the Ketton building stone. Being the transition between a pure siliceous sand and an essentially pure calcareous limestone, the stone is a mixture of both materials. As can be seen in **Figure 1** the stone consists of a mass of very fine quartz sand grains, typically between about 75 microns and 90 microns in diameter, set in a matrix of interlocking calcite crystals, typically about 500 microns in diameter. Small quantities of mica are sometimes present and it has been suggested that these may play a part in producing the fissile nature of the stone. Although the stone can be cleaved into thin layers when it is found at outcrop, the unexposed stone, or 'log', when extracted in the traditional manner from shallow mines, is found to be in the form of a relatively massive bed.

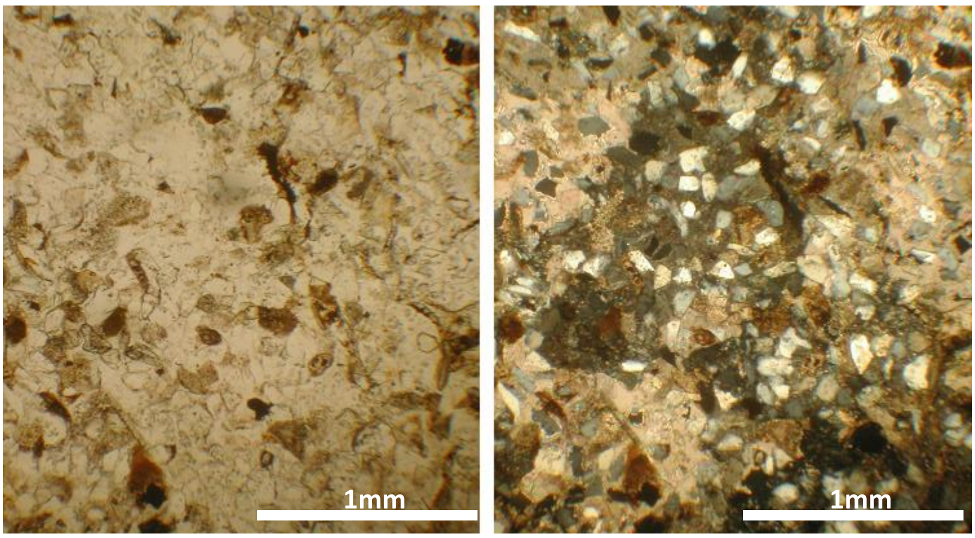


Figure 1 Photomicrographs of a representative sample of Collyweston 'Slate'. The photograph to the left, taken in plane polarised light, shows the clear grains of quartz in a calcite matrix, which under crossed Nicols (photograph to the right can be seen to be a complex of large calcite crystals, each enclosing a number of quartz grains. The brownish darker material at the top of the photographs is limonite.

The surfaces along which the stone splits are sedimentary depositional surfaces. As previously mentioned, the stone has not been metamorphosed therefore the term ‘slate’, although used locally, is technically incorrect, the roofing material produced at Collyweston and surrounding areas being stone roofing tiles (tilestones). Being depositional surfaces there tends to be a slight variation in the petrography at the interface between each of the lamina. Flakes of mica tend to concentrate at these points, although never in sufficient quantity to produce an obvious weakening due to the accumulated cleavage of the grains. There is often a slight increase in the quantity of limonite at the laminations, suggesting there may have been a slight increase in organic matter at that point. There may also be a slight increase in porosity at the laminations, although this is scattered. Although the mica, the iron mineral and the porosity may result in weaknesses at the laminae, this is countered by the fact that the large calcite crystals which make up the body of the stone can pass across the laminae unaffected by their presence. This may be the reason why the cleaved surfaces of the tilestones tend to be relatively rough and irregular, the calcite crystals having to be sheared when the stone splits.

Stone Characterisation

X-ray diffraction (XRD) patterns were obtained from powdered samples using a Philips X'Pert Pro diffraction system equipped with an ‘Accelerator’ detector and a Cu-tube (λ = 1.542 Å), operating at 40 kV and 40 mA. The angular range of 5 to 15 °2θ was also investigated using a mini-proportional detector, but no discerning peaks were detected. Data was collected using the X'Pert Data Collection software and diffraction data analysed using X'Pert Highscore Plus software and standards library database to identify the components present.

Fourier transform infrared spectroscopy (FTIR) analysis was performed using a Thermo Nicolet Nexus spectrometer and a Graseby Specac ‘Selector’ DRIFTS accessory. Samples at 5 wt% were ground with KBr for 1 minute using a pestle and mortar. Spectra of sample and KBr mixtures were ratioed against that of KBr alone. X-ray fluorescence (XRF) was performed using a Philips MagiX Pro spectrometer and UniQuant analysis software and using beads formed by fusing the sample with lithium tetraborate.

Scanning electron microscopy (SEM) used an FEI Quanta 650 SEM with tungsten electron source coupled with Oxford Instruments AZtec Energy Dispersive Spectroscopy (EDX).

Results

XRD (**Figure 2**) and FTIR analysis (**Figure 3**) confirmed the dominant presence of quartz and calcite in both the buff and blue areas of the stone, whilst elemental composition determined by XRF showed a slightly higher Ca and lower Si content in the buff area (CaO = 25.0% and SiO_2 = 46.8%) compared to the blue area (CaO = 21.0% and SiO_2 = 56.0%) indicating, respectively, higher and lower quantities of carbonate and quartz. Although only a 10 g fraction from each area was analysed it will not fully represent every area within a mine or even a log, however, it does show that the elemental composition varies significantly within the stone.

The more intense kaolinite bands (four characteristic bands between 3695 and 3620cm^{-1}) present in the infrared spectrum collected from the blue area compared to that obtained from the buff area indicates a higher quantity of this mineral. This is supported by the higher Al_2O_3 content in the blue area determined by XRF (3.06 compared to 1.79%) even though there is a contribution to the Al_2O_3 content from chlorite, which is also present in the blue region as evidenced by characteristic infrared bands at 3549, 3492 and 3402 cm^{-1}.

SEM images obtained via a low magnification (**Figure 4a - x250**) from a 'naturally' cleaved surface exhibits fractured and non-fractured grains together with groups of smaller particulate coverings. The dominant larger grains (50 to 250 μm) contain only calcium and oxygen (as determined by EDX analysis) and are therefore assigned as calcite; some of which (**Figure 4b**) are porous in nature. The larger grains of 500 μm observed in the petrography results may have undergone fracture or be present as whole grains but only a portion of them are observable, the hidden portion being submerged under the fractured surface. Alternatively, it is possible that the cleavage surfaces represent a period of low-speed currents which only deposited grains of phyllosilicate and very small grains of quartz, the coarser grains seen in thin section being in the body of the stone formed when deposition was from faster moving currents. The smaller quartz particles range in size from a few microns to approximately 20 μm, which correlates with those observed in the photomicrographs (**Figure 1**). The quartz particles appear to cover the calcite grains and line pores formed between grains (**Figures 4c and 4d**). Similar coverings are observed by clay minerals, which are characterised by their smaller platy morphology and silicon and aluminium elemental content (**Figures 4e and 4f**). The morphology is not distinct enough to determine whether the clay minerals are either kaolinite or chlorite. Interestingly, there are particulate areas rich in aluminium and silicon alongside discrete, but closely connected particulate areas rich in titanium (**Figure 5**) and coating calcite grains. Elevated levels of titanium in such areas may be indicative of the initial formation of a mineral such as odinite $(Fe,Mg,Al,Fe,Ti,Mn)_{2.4}((Si,Al)_2O_5)(OH)_4)$, which can be found in recent sediments as infilling of biogenic or detrital porous grains in tropical latitudes (Bailey, 1988) and are usually altered to chlorite in older sediments (Worden,2009).

The small lining particles (both quartz and water swelling clay minerals) occurring between the larger and sometimes porous calcite grains could be envisaged to offer weak points between contacting calcite grains and thus a mechanism for which cleavage could occur during the expansion of water during freezing, especially if concentrated at the cleavage sites. Titanium and oxygen rich phases were also observed in the absence of aluminium and silicon and based on their morphology are likely to be rutile. In addition, some larger grains are thought to be potassium-feldspar as evidenced by containing potassium, aluminium and silicon.

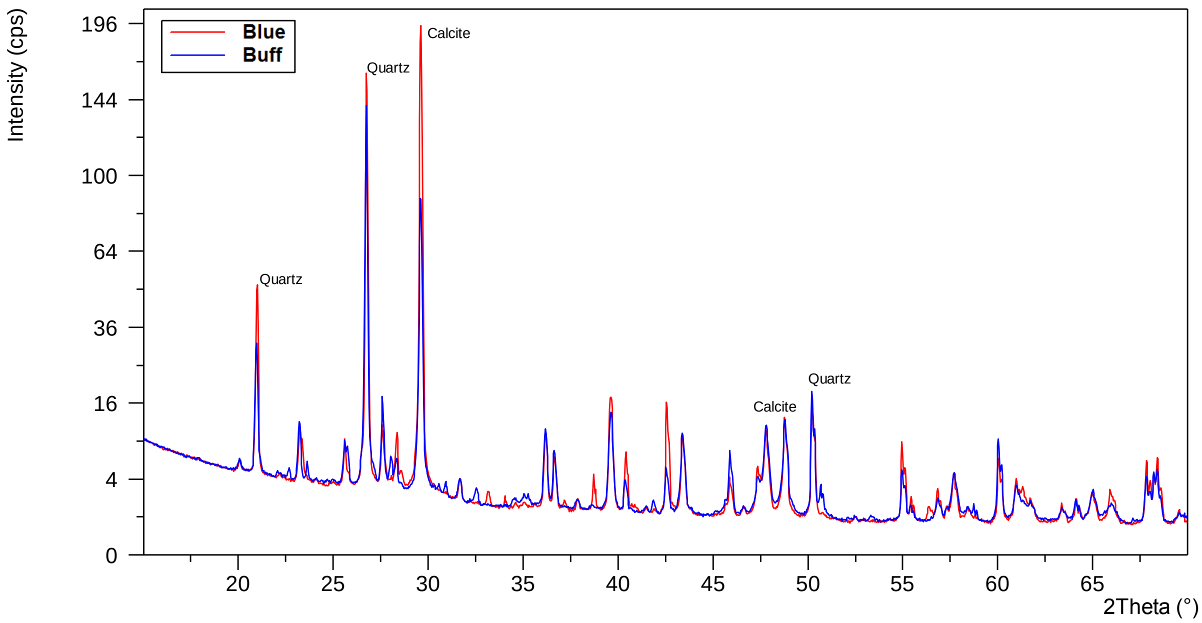


Figure 2 XRD traces collected from the blue and buff areas of Collyweston stone, the main characteristic peaks of quartz and calcite are identified.

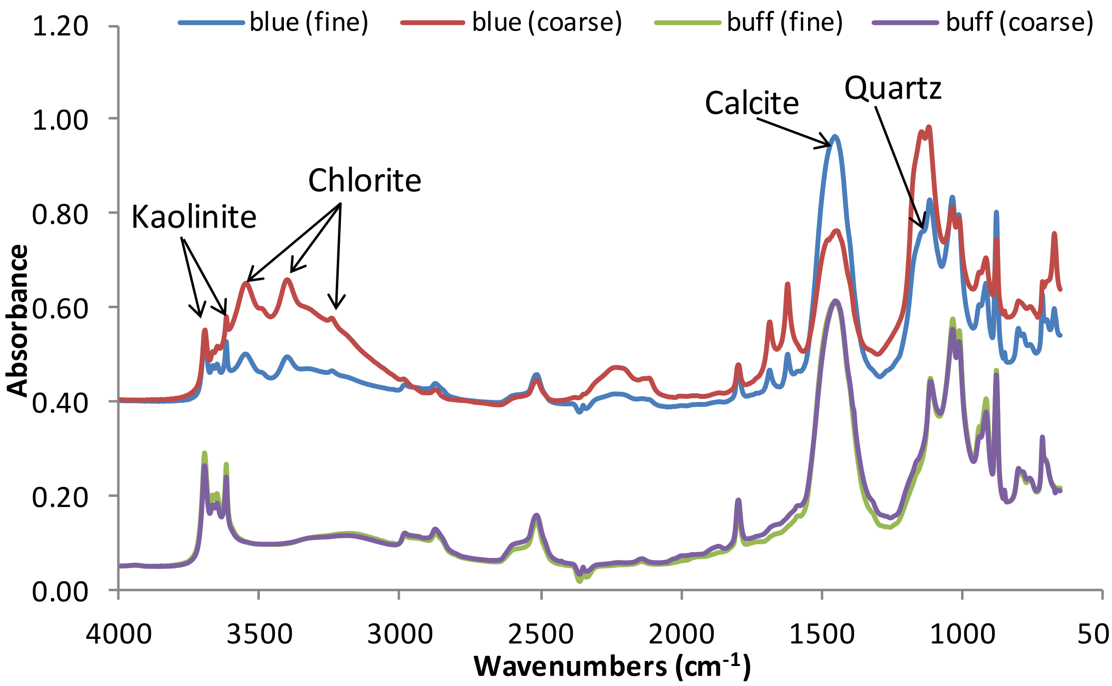


Figure 3 Infrared spectra collected from the coarse and fine fractions of blue and buff Collyweston stone. Fine and coarse grains were obtained from the suspended and sedimented fractions, respectively, taken after lightly crushing the stone with a pestle and mortar, placing in deionised water, ultrasonication, shaking and standing for a few seconds.

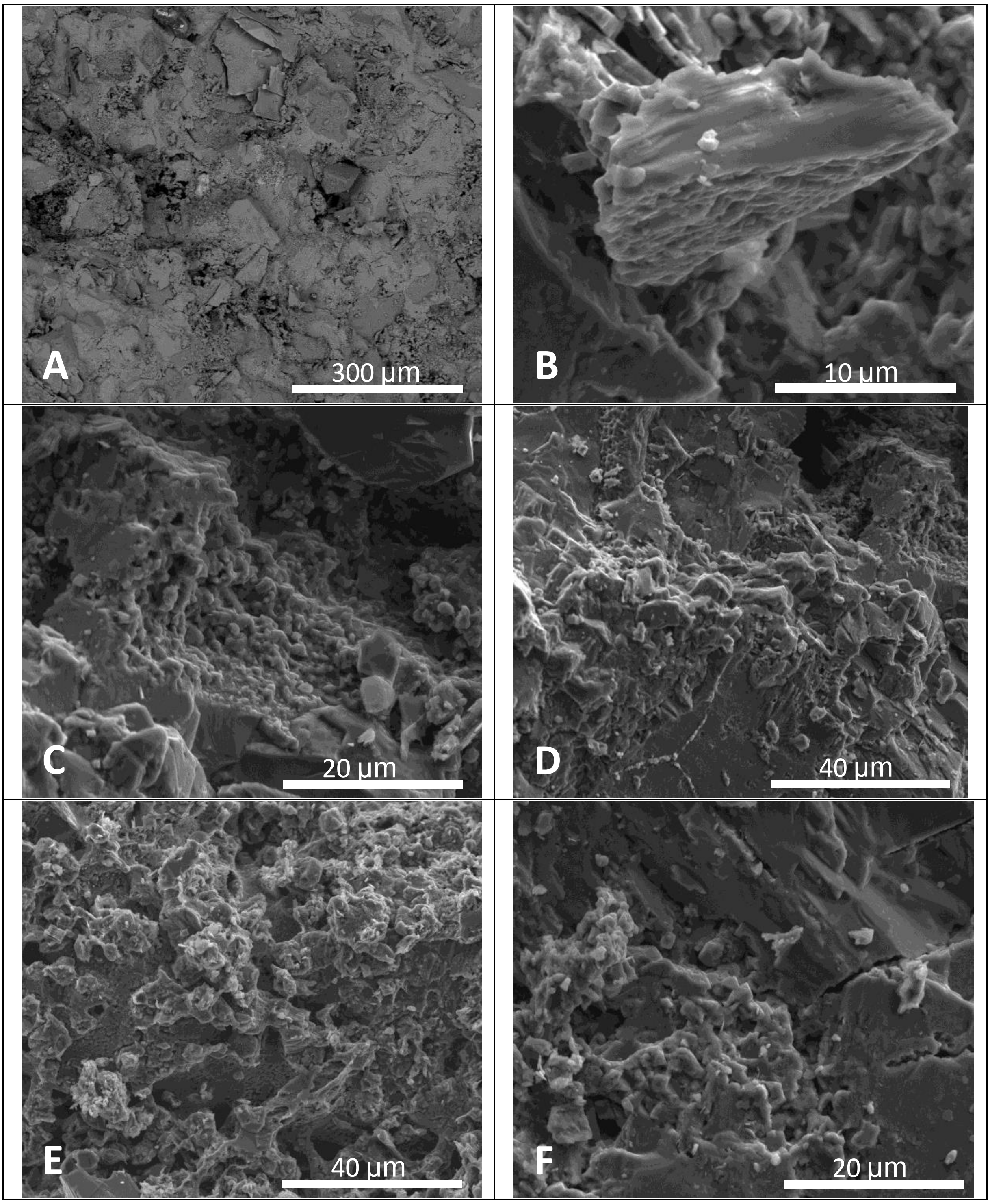


Figure 4 SEM images obtained from naturally cleaved Collyweston stone. a) BSE image, b-f) SE images.

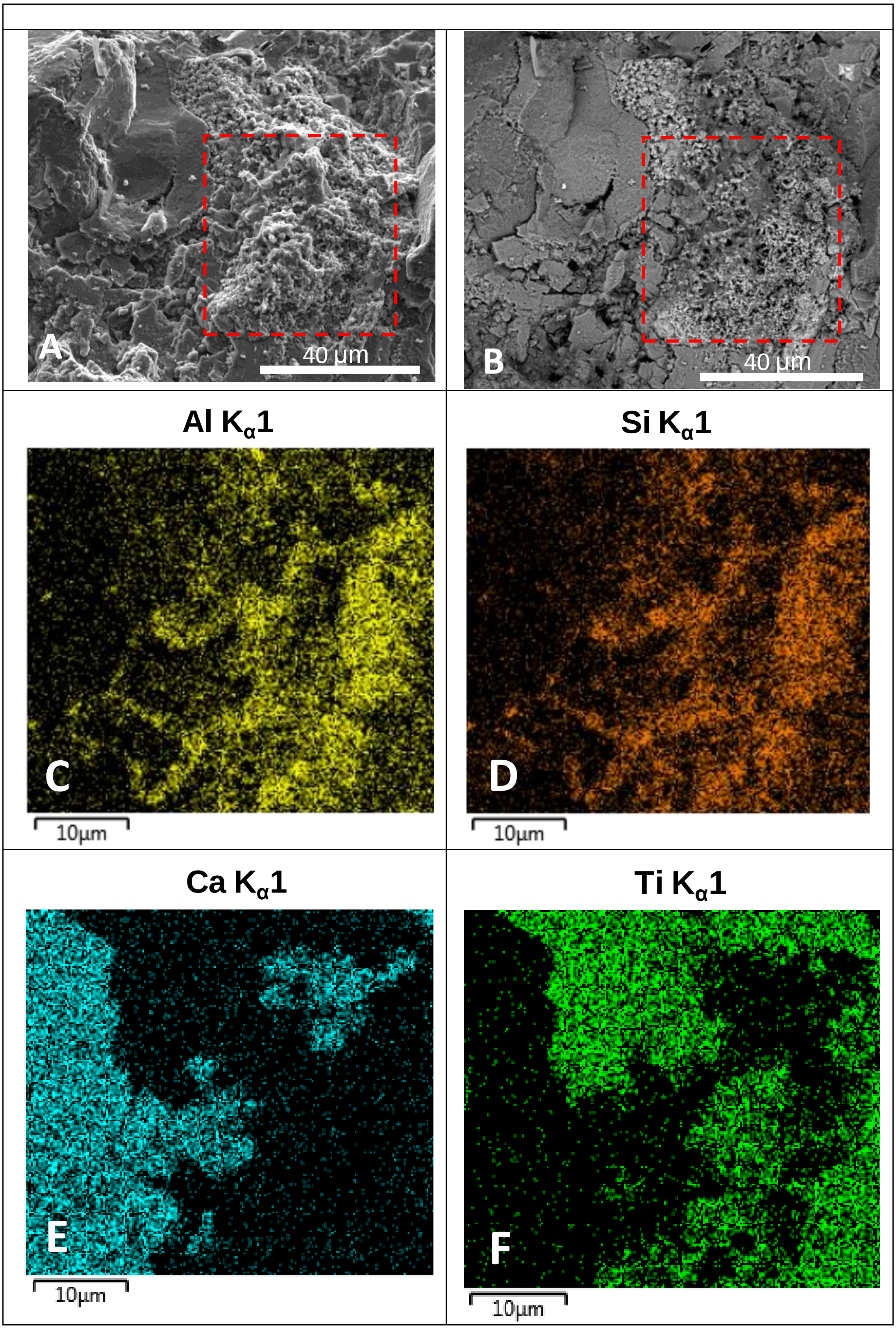


Figure 5 SEM images obtained from cleaved Collyweston stone, a) SE image, b) BSE image, c-f) elemental maps obtained from area highlighted by red dashed square.

Phases 1 & 2

The initial work on artificially producing tilestone from the limestone was undertaken for the Burghley Estate in 1998, in order to test the suitability of the limestone samples for accelerated frost splitting. The test chamber used for the work was 1.9 m by 2.0 m on the base and 1.2 m high (external dimensions) with the outer skin having 80 mm of insulation between external (ambient) and internal conditions. This chamber was modified by SHU to facilitate computer control of the cycle such that any period of freezing, thawing, simulated raining or draining could easily be performed and with finer control over the number of cycles per day and the maximum and minimum temperatures (Laycock , 2002; Laycock & Yates 2000). The duration of the component periods of the freezing and thawing cycles can all be altered although generally the cycle consists of four discrete elements; a freezing cycle, a period of thaw, a period of rain and a period of drain which allows excess water to exit the chamber before the next freezing cycle begins.

Figure 6 Material from the base of the limestone being removed from a mine for testing.

The logs chosen for phase 1 of the study were collected directly from the mine (**Figure 6**) and therefore contained their natural water content. This was maintained by wrapping in waterproof material on site before being transported to the laboratory. On arrival all samples were immediately placed in conditions of high relative humidity (80%) in an attempt to retard the adverse drying effects mentioned in the literature review. For Phase 2 of the study the logs were stored under water prior to transporting back to the laboratory for testing in order to prevent the samples drying out. Frosting was based on previous work undertaken on brick and stone (Laycock, 2002; Laycock& Yates, 2000) and with initial reference to the CERAM test (British Ceramic Society, 1984), which is referred to in BS 3921:1985, BS 5628;2005 and BS EN 771:2003 and 2011 and later detailed in DD CEN/TS 772-

22:2006. The requirements of the frosting were that sufficient delamination should be generated to allow the stones to cleave without this becoming so intense as to cause any extensive loss of materials or to jeopardise later frost resistance of the tilestones produced. **Table 1** shows the variations in temperature maximum and minimum with the initial regime used being the British Ceramic Research (1984) cycle. Relative humidity in the BCRL chamber is not controlled but logging confirms that it remains above 60% during the initial stage of freeze-down and at an average of 80% throughout the cycle.

	Phase 1						Phase 2						
Test no	1	2	3	4	5	6	1	2	3	4	5	6	7
Maximum temperature (°C)	+25	+10	+10	+10	+10	+15	10	10	10	10	10	10	10
To minimum temperature (°C)	-15	-5	-5	-5	-5	-5	-5	-5	-5	-5	-5	-5	-5
Minimum Freeze Rate (°C/min)	0.33	0.13	0.13	0.13	0.13	1.5	1.5	3	5	10	3	5	10
Residence at lowest temperature	Y	Y	N	N	N	Y	Y	Y	Y	Y	Y	Y	Y
Cycles /day	10	1	3	4	8	3	1.5	3	4	8	2.2	3.3	6.5

Table 1 Summary of temperature regimes used for the Phase 1 and 2 work (13 tests, 1 log sample used for each test)

Observations were made on the nature and extent of splitting, whether tilestones of suitable thickness were able to be produced without unnecessary manual effort, and whether these were of suitable quality to warrant further investigation. The aspiration was to identify a cycle regime which enabled an economic turn round of adequate splitting with the freeze/thaw cycle at a temperature which would not require excessive energy demands.

Phase 3

For phase 3 of the study the material used was borehole core recovered as part of the investigation into the resource available on the Burghley Estate. This investigation was undertaken to establish the number of splits which could be generated from log in different locations throughout the area of the deposit and thus allow the most economically feasible stone to be identified. The borehole drilling itself was located in an area which had been identified by a Ground Penetrating Radar survey as not having been previously exploited by mining. The area of potential resource initially investigated is shown in **Figure 7** and the area around Collyweston known to have been exploited in **Figure 8**. The exploration drilling programme involved nineteen diamond cored boreholes. All of the 100 mm diameter core obtained was used in testing, an example being shown in **Figure 9**.

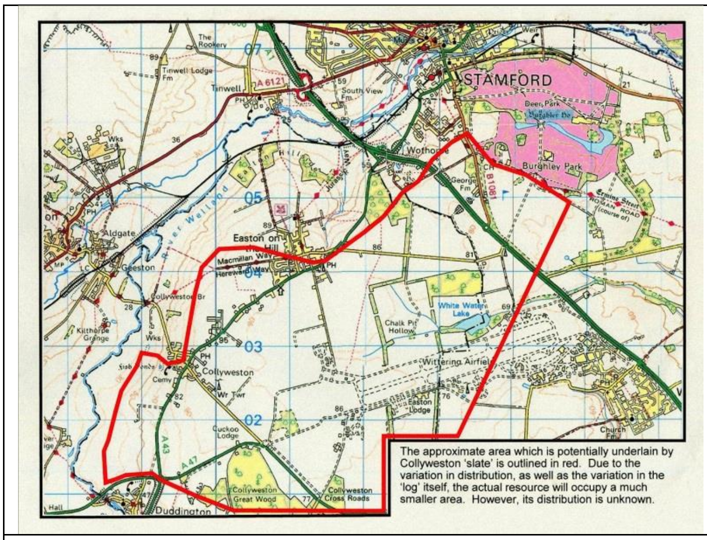


Figure 7 Map of the area to the south west of Stamford showing the limits of the area under investigation.

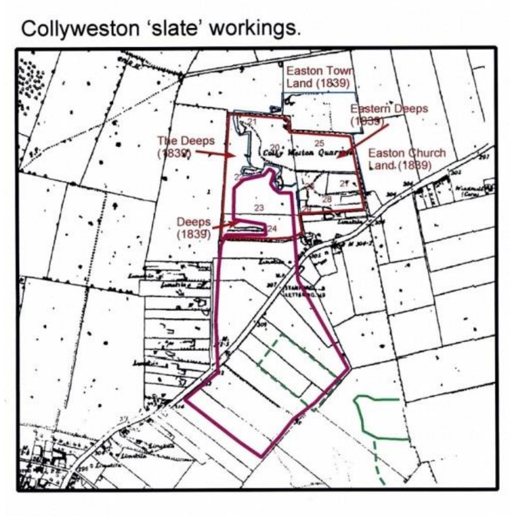


Figure 8 Map of old limestone workings around Collyweston

Figure 9 Composite photograph of a typical section of Collyweston Limestone borehole core

Phase 4

Having established that a borehole core could be easily split under laboratory conditions, it was necessary to translate these results into practical block sizes and commercial quantities. A 40 ft. mobile freezer unit, of the type used to transport frozen goods, was therefore utilised as a pilot plant. Various modifications were made to the configuration of the pilot plant and the operating cycle as the tests progressed. The aim of the pilot plant was to reproduce the natural cooling and warming cycle of a winter's day which resulted in the development of frost. It was also known that the stone has to be kept wet, since if allowed to dry out freezing does not result in effective splitting of the log. During the initial tests the log was kept wet by storage in an agricultural water tank and then placed in the freezer. Unfortunately, due to the fact that the storage area within the freezer is kept cold by recycling the air within the unit through the freezer coils, a process which condenses moisture in the air and deposits this on the coils. The unit soon failed due to the freezer plant coils being solidly encased in ice. If this type of freezing unit is to be used, that is one where the freezing air passes around the stone, it is necessary to develop some form of system whereby the stone is kept moist but the air is dry. Relative humidity in the pilot plants could not be controlled and excessive moisture condensed on the cooling coils leading to loss of efficiency. The problem was solved by placing the logs in individual large plastic bags to seal in the moisture while allowing the cooling air to maintain its low humidity. It is important that the plastic bags make as close a contact as possible with the stone, so as to ensure there is no layer of stationary air within the bag which would act as an insulator and reduce the cooling effect of the external cold air. Previous work had demonstrated that the rate of cooling was important, as was the rate at which the minimum and maximum temperatures were achieved. It was considered that the optimum conditions would be based on the size of the freezer, the number and sizes of the logs and the design of the air flow around the log. Tests had to be carried out to determine all these parameters. One important measure during the tests was power consumption, since this is probably the most expensive part of the operation. The size of the log is limited by the ease with which they can be extracted, brought up a mine shaft, wrapped, palletised and placed in the freezer. The normal thickness of the bed used to form logs is 900 mm, sometimes a little less. The top and bottom of a good log are parallel. Using a standard pallet, the log size was therefore limited to about 900 x 1,000 x 1,000 mm. The temperature within the plant was kept within those which would be encountered by a log when frozen naturally, in the traditional manner, in January and February. Using freezing temperatures which were in the range of -5 or -10°C and thaw temperatures a little over 0°C the aim was to prove that the production of tilestones was able to be scaled.

Phase 5 Apethorpe

Following the conclusion of the Phase 4 testing and a successful mining feasibility study undertaken by a firm of international mining consultants, Burghley Estate made the decision not to proceed further at that particular time with the development of a production plant for reasons not associated with the practicalities of the project. However English Heritage (now Historic England) who had been following the progress of the studies, had an urgent requirement for Collyweston stone roofing for major conservation work at Apethorpe Palace. Following discussions with Burghley Estate, permission was granted for Historic England to install a small freezer unit at Apethorpe, in order to produce new tilestones for the building. Since the freezer to be used was smaller and of a different design a further development phase was required, to optimise the operation of this unit.

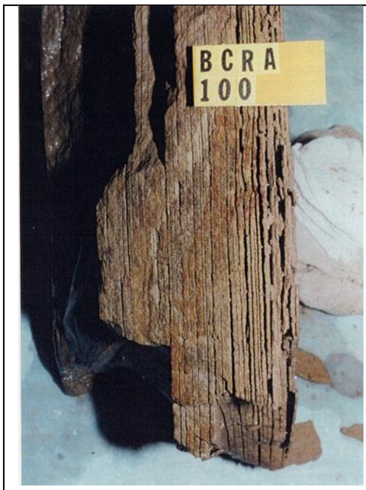
Figure 10 Stone logs in outdoor store at Apethorpe.

Messenger Construction Limited, who were already carrying out conservation work at Apethorpe, were instructed by Heritage England to oversee the day to day running of the production test chamber at Apethorpe (Morell, 2013). The chosen log for the first trial had been previously stored exposed to the elements (**Figure 10)** and was considered to have dried. It was therefore stored under water for 5 days prior to freezing. In order to prevent the cooling coils in the freezer from icing, the log was placed on a neoprene mat and wrapped in plastic before being placed on a plastic pallet for transport to the freezer unit. Freezing was initially performed on a 4 hour cycle of -8ºC to +8ºC and cycled for 17 days. The second trial used a lower temperature regime (-20ºC), and the third trial used fresh material collected from Bullimore's Quarry at Duddington, with four logs being cycled immediately after collection and the remaining five logs after being stored under water using a regime of -8ºC to +8 ºC on 4 hour cycle initially which was later extended to an 8 hour cycle. Each cycle was such that half the time was spent in freezing mode and half in thawing mode, The limitation of the freezer unit used was that there was no control over the rate of freezing, To ensure that the logs were experiencing freezing temperatures at depth thermocouples were inserted into the stone.

8. Results and Discussion

Phases 1 and 2

The first two phases of the work indicated that samples experiencing unrealistically harsh freezing conditions using the BCRL cycle (+25 to -15 ºC) generated extensive delamination (e.g. Table 1 phase 1 Test 1; **Figure 11a**) to such an extent that it was unusable. Realistic freezing temperatures and temperature regimes such as Test 2 in phase 1 (Table 1) required the use of a bolster to fully separate the stone after delamination had occurred (early phases are shown in **Figure 11b**) and tilestones produced were too thick (**Figure 11c**). Thus there are optimum conditions which must be achieved where splitting occurs with minimum wastage and within a short period of freezing; this was deemed to be Tests 3 and 5 from the phase 2 trial (Table 1). As the phase 1 testing took several months to accomplish, the log was stored in a humidity controlled environment as well as being kept wrapped in waterproof materials in an attempt to maintain the as mined condition. Although an attempt was made to prevent dehydration prior to testing the stored stone it appeared to be less susceptible to splitting than the stone which was tested 'fresh' from the mine. As a result of this observation the phase 2 stone was therefore stored under wateruntil testing could be carried out..

<table>
<tr>
<td>
</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Figure 11a Sample K-09 showing extreme delamination damage at end of cycling with BCRA cycle.</td>
<td>Figure 11b Log K10 After 100 cycles of freeze/thaw. Early phases of delamination.</td>
<td>Figure 11c Log K10 after 100 cycles freeze/thaw and application of mechanical splitting effort. Note excessive thickness of tilestones formed.</td>
</tr>
</table>

The material collected for phase 2 of the work was much more variable in colour than that for phase 1, and the samples were larger in size. There was a distinct difference in the apparent weights of samples of similar size indicating that different densities and porosities might be present. Of the six samples, half were blue hearted (buff exterior, blue interior), and half were buff stone. Testing showed that the blue hearts to the material are of much lower apparent porosity and higher density than the buff material leading to variability in the frosting performance as a result of differential water intake in the two material types; the buff material splitting more readily than the blue hearted. The time required to achieve freezing to the core of the log is a function of its size, thermal mass, thickness (smallest dimension), and also the lowest temperature and duration of the freezing period.

All these parameters will affect the number of cycles required to cause splitting and the optimum was found to be 10 on the small samples initially tested, assuming that the samples have been stored in a saturated state. In larger samples, more cycles were therefore needed to cause the same level of delamination required for easy manual splitting. It was found that ice forms at the centre of a log of mass 9755 g approximately 85 minutes after the start of the freezing cycle, i.e. approximately 70 minutes after the temperature passes 0ºC.

Phase 3

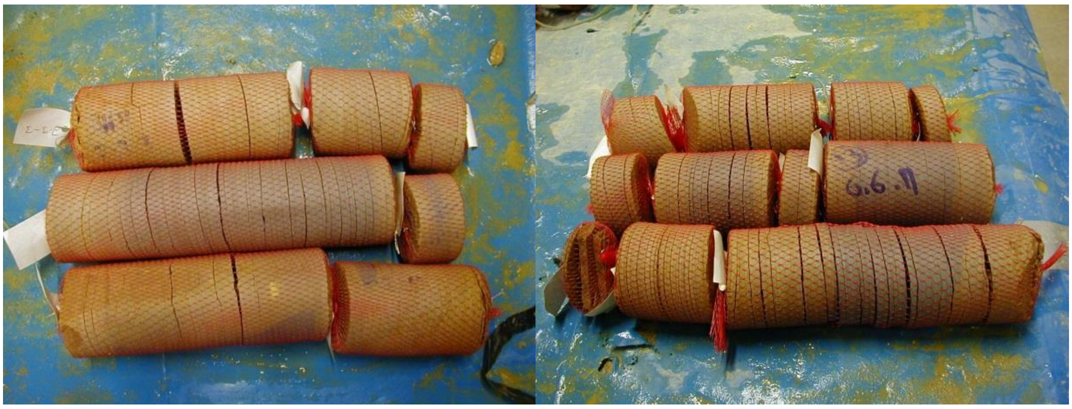


Figure 12 Typical progression of splitting at 10 cycles (left) and 20 cycles (right)

The test work on the recovered borehole core demonstrated that the required thicknesses of tilestones (**Figure 12**) could easily be obtained on the fresh material recovered from the area under investigation. The splits were continuous, needing no further mechanical effort. The mining study concluded that the new mine was economically viable and that it would be possible to utilise mechanical mining techniques to extract the log (**Figure 13**). A freezing unit could therefore be operated, if proved, on a continuous basis resulting in a cost-effective splitting process.

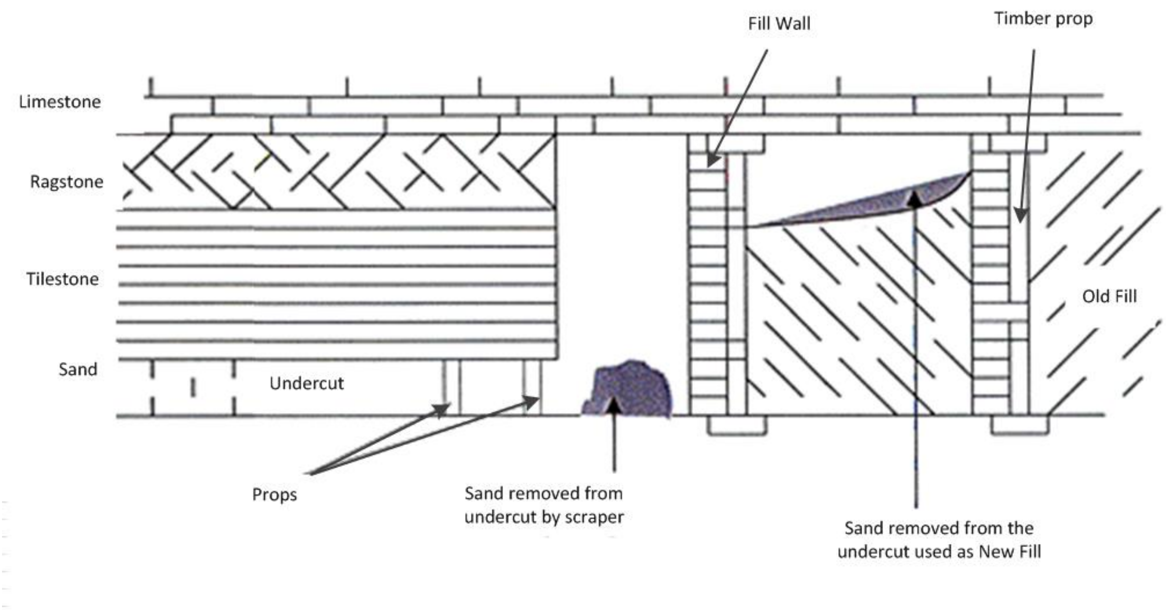


Figure 13 Underground methods of extraction (Diagram modified after SRK Consulting report of 2004)

Phase 4

Parameters of the protocol (including temperature range and rate of freezing) have to be optimized according to the stone slab mass and geometrical characteristics to ensure the effectiveness of further splitting Due to material losses the log extracted to be formed into tilestones will need to be bigger in order to form the larger finished tiles required. These would require more cycles of frosting. The pilot plant demonstrated that the variability in size, configuration and the number of pieces of log and the air flow around the blocks changed the efficacy of the process. Power consumption was measured and the cost of electricity for the pilot plant used for the work was approximately £8.50 / day over the trial period of some 80 days. The optimised regime provided splitting of log sufficient to produce approximately 3.5 roofing squares within 2 days at 8 cycles per day.

Consistency can be improved by closer control of log size extracted and limiting as much as possible the volume of poorer quality log. The top and bottom of good log are parallel with internal laminations parallel to these surfaces. Where the base of the log is rounded due to the limestone filling a channel or a scoop in the underlying sand the feature may or may not split. Using a standard pallet, the log size is limited to about 900 (h) x 1,000 x 1,000 mm. Storing the log under water and then placing in sealed high density plastic bags immediately on arrival at the pilot plant and before being placed in the freezer unit was found to increase the efficiency of the splitting process. Ducting the air within the chamber also improved the consistency of the regime within the unit. The pilot plant used for splitting

Collyweston 'log' by freezing and thawing regimes between +8 and -8°C with between 4 and 8 cycles per day has indicated that the process can be achieved on a continuous basis when using non-specialist equipment. Unlike the results using the borehole core, the pilot plant did not necessarily produce visible continuous splits in the 'log'. However, even where no physical splits had been produced, splitting along laminae in the stone could be easily achieved with a hammer and chisel. It was noted that the quality of much of the material used throughout most of the pilot trial had not been based on the type of stone which would normally have been selected underground as suitable for frost-splitting. The last batch was extracted from the old Burghley Pit (The Deeps) and was of much better quality, producing visible splitting quite readily. Even with the poorer quality stone, the pilot plant was able to produce on average 3.5 square metres of roofing material as laid every 2 days. Subject to the costs of mine development and freeze-thaw plant construction and subject to planning permission the 3,000 roofing squares of Collyweston Slate identified in the area investigated by borehole drilling could be converted into a proved mineral resource.

Phase 5

Messenger Construction Limited was instructed to oversee the day to day running of the production test chamber at Apethorpe and to produce a report (Morell, 2013). The chosen log had been previously stored exposed to the elements for over a year and was considered to have dried (**Figure 10**). Freezing was initially performed on a 4 hour cycle of -8ºC to +8ºC and repeated for 17 days. While the log split evenly the thicknesses could only be reduced to between 100 and 150 mm. In an attempt to split the stored stone a second trial with a lower temperature regime decreasing to -20ºC and a four hour cycle was tried but also found to be ineffective. The third trial therefore used fresh material collected from Bullimore's Quarry at Duddington, with four logs being cycled immediately after collection and treated for 6 days. Log used in the first trial initially split into 100mm to 175mm thicknesses. The logs were reduced in size and put back in the freezer unit for a further 2 days to achieve split thicknesses of 50-100 mm. After a further cycle some usable slate of approximately 15mm was obtained. The five logs obtained as fresh material and stored under water were cycled using a regime of -8ºC to +8 ºC on 4 hour cycle initially for 6 days. Thermocouple monitoring indicated that the larger log did experience freezing in the centre during the regime. While this could be made to split it was found that log sizes of 600 mm square were both easier to handle and split with greater ease. The log sizes were reduced to 600 mm square which was consistent with estimated sizes from the historical photograph (Plate VI) in the Moreton in Marsh Memoir (Linsdall, 1929).

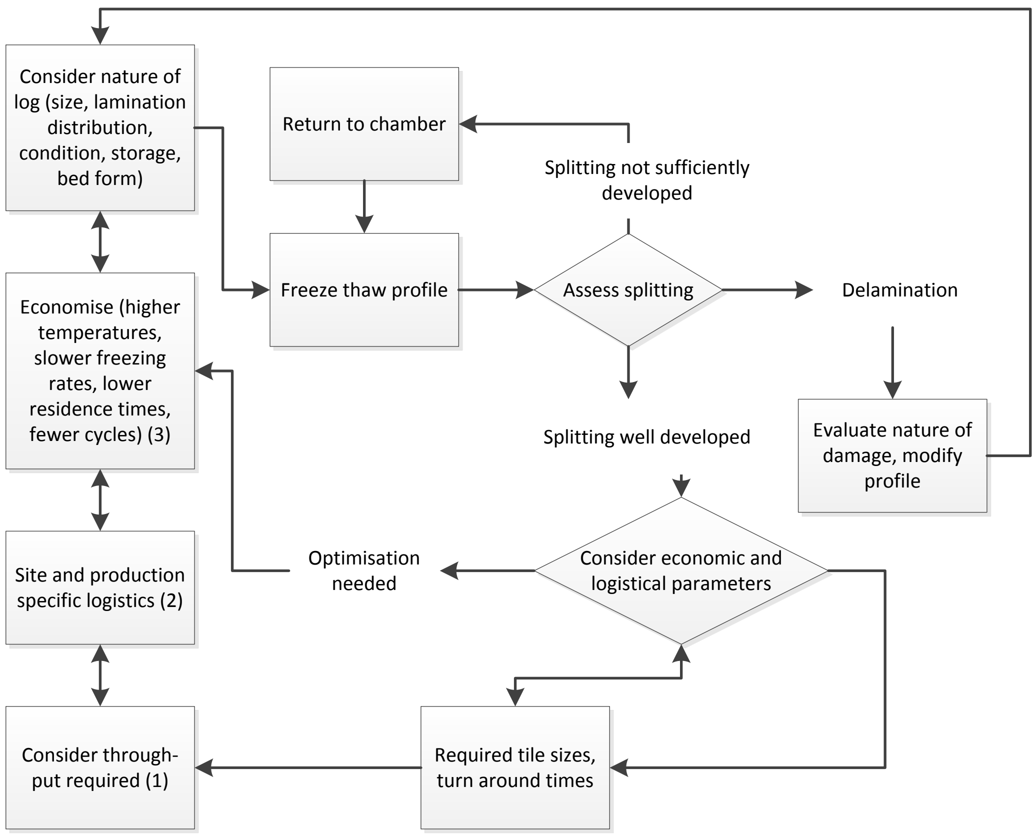
The second pilot plant work concluded that initial frosting followed by manual splitting and return of material to the chamber for further frosting was capable of producing useable tilestones. The second pilot plant was deemed to be less effective than that of the phase 4 study, which had vents in the floor to allow the cold air to pass up round the logs and achieve a uniform air flow. In the smaller freezer unit used at Apethorpe, the air entered at the base of the end wall adjacent to the freezer unit. This made it necessary to install a polythene tunnel to ensure that the air flowed past all the blocks to the door end of the unit. It is considered that floor vents are probably a more controllable system. Even so, the process was successful and log was split in a manner which enabled the result to be dressed as tilestones (**Figure 14**).

Figure 14 Tilestones produced by Messenger Construction Ltd at Apethorpe

As mentioned above the log produces good tilestone when the top and bottom of the log are parallel, and all the internal laminations are parallel to these surfaces. However the natural material varies both vertically and laterally. Sometimes it is difficult to see many laminations and in other instances the laminations are irregular or wedge shaped. Although such material may split to a certain extent, the resulting tilestone will be wedge-shaped and not really suitable for good quality roofing material. The reason for the dissimilarity is due to differences in sedimentation. The uniform stone, containing many thin parallel laminae, was deposited in unchanging conditions away from fast currents or channels. The more irregular material formed in or near channels in faster flowing and more irregular currents. This variation was noted during the traditional extraction down the mine and only the good quality material was brought to the surface, perhaps 50% never seeing the light of day.

Summary

The overall aim of the work was to develop a method by which the Collyweston stone roofing 'slate' could be commercially produced throughout the year. To do so it was necessary to demonstrate that artificial freeze/thaw weathering was financially viable, and to determine the limits of temperature, and the rate of change of temperature, within which the equipment and the operatives should normally be working. There were a number of constraints to the work, the foremost being that stone is a natural material, with petrographic properties which vary continuously, resulting in such physical properties as thermal conductivity, porosity and permeability also varying. In addition, the ex-mine blocks will vary in size and irregularity. Unlike other building materials such as cement, lime and brick, there is no processing of uniform raw materials. The splitting of the log into tilestones required process which could be carried out in the vicinity of the mine, by the operatives who also select and extract the stone underground without professional or laboratory backup. Different batches of log from different parts of the mine may therefore require different freeze/thaw regimes. This ultimately will be assessed by the operatives using visual appraisal of the raw material and experience.



1) Higher through-puts could justify additional chambers. For high volumes a double system would allow the exhaust gases from one chilling one chamber to aid defrosting in a second chamber with additional cost savings. For smaller productions a single chamber run on a batch basis would be more cost effective.
2) Loading and unloading of the chamber needs to be co-ordinated as closely as possible to production such that the natural moisture levels in the stone are not lost. A contingency for freezer plant failure needs to be considered
3) The chamber should be internally flexible in order to cope with larger or smaller batches as log is produced. Log should be split as soon as possible after mining and not stored. The freezer unit must also be designed to maintain the same air flow and temperature changes whatever the volume of material being processed.

Figure 15 Summary of the factors which need to be evaluated for successful production of collyweston 'slate'

9. Conclusions

Extraction of the limestone roofing slate from Collyweston was an industry which was largely considered extinct, with building repairs relying on wholesale recycling of roofing from demolished buildings. There are few sources of literature which describe traditional extraction processes despite the historically significant volume of tilestones used. This work was undertaken to determine if it was feasible to revitalise the industry and artificially replicate the natural freeze/thaw conditions which historically have be used to split Collyweston log using low-technology equipment. When the means of splitting was proved possible it was intended to determine by experiment, the optimum physical configuration of the processing unit to replicate those conditions. Experimentation confirmed historical references to the importance of the maintenance of the natural moisture in the stone as an important factor. Work was undertaken over several years to investigate the resources available and whether these could be artificially frozen to produce slates. The work identified a freeze/thaw regime which could be used to produce roofing slates for historic buildings and which were used in the English Heritage (now Historic England) restoration of Apethorpe. Initial investigation work using petrographic and electron microscope studies attempted to determine the process by which the splitting occurs, however this proved inconclusive and further work will be required in order to understand the mechanisms by which facilitate some but not all of the laminae to split during freezing and thereafter stabilise. Additionally, if the stone is allowed to dry out, the splitting mechanism is destroyed. Under perfect conditions, the 900 mm thick log will split into about 50 sheets. The number of laminae in the stone is greater than this but splitting only appears to occur along some of these, about 1 in 20. Even after frosting, manual splitting is required. Since a naturally variable material is utilised (stone) it would be very complex to produce the perfect freezing regime for a large selection of logs. The operation of a production freezer unit would need to be based on a combination of science and experience built up by the operator and predominantly based on log size, texture and number and quality of laminations. The experienced operator would need to adjust the cycle times and number of logs in the freezer unit to suit each particular batch of log.

10. References

Ashton, M 1980. The stratigraphy of the Lincolnshire Limestone Formation (Bajocian) in Lincolnshire and Rutland (Leicestershire). Proceedings of the Geologists' Association, Vol.91, 203-224.

BAILEY S. W. (1988) Odinite, a new dioctahedraltrioctahedral Fe3+ rich 1:1 Clay Mineral. Clay Minerals 23, 237-247 http://www.minersoc.org/pages/Archive-CM/Volume_23/23-3-237.pdf

BGS (2016) The BGS Lexicon of Named Rock Units Lincolnshire Limestone Formation "British Geological Survey 1998 - 2016 (c)NERC http://www.bgs.ac.uk/lexicon/lexicon.cfm?pub=LL

British Ceramic Society 1984 Transactions and Journal of the British Ceramic Society, Vol. 83 (1984 British Ceramic Research Association, Queens Road, Penkhull, Stoke-on-Trent ST4 7LQ.

Collyweston Stone Slaters' Trust, 2001. Collyweston Stone Slate. A Guide. www.collywestonstoneslaterstrust.org.uk

Cox, N (2009) Belton Boathouse, Journal of Architectural Conservation, 15:2, 7-26, DOI: 10.1080/13556207.2009.10785045 To link to this article: http://dx.doi.org/10.1080/13556207.2009.10785045

Goodwin J M, 1987. The Book of Collyweston. Spiegl Press, Stamford. Pp 55-58

Harding, S C. 2014 Ichnology, Mineralogy and Palaeoenvironmental implications of the verdine and glaucony facies in sedimentary rock. PhD Thesis. Department of Geology and Geophysics. The University of Utah. December 2014

Hesselbro, S. (2008) Sequence stratigraphy and inferred relative sea-level change from the onshore British Jurassic. Proceedings of the Geologists' Association, 119,19-24

Historic England 2005 Stone Slate Roofing Technical Advice Note. London:English Heritage Published 14 October 2005

Historic England 2011 Strategic Stone Study. A Building Stone Atlas of Northamptonshire. Published August 2011 https://www.bgs.ac.uk/mineralsuk/buildingStones/StrategicStoneStudy/EH_atlases.html

ICS 2013 International Commission on Stratigraphy Chronostratigraphic chart v2013/01 http://www.stratigraphy.org/icschart/chronostratchart2013-01.pdf

Judd, J W. 1875. The geology of Rutland and parts of Lincoln, Leicester, Northampton, Huntingdon and Cambridge. Memoir of the Geological Survey of Great Britain (Old Series sheet 64) p140, 182

King, A. 2010 Northamptonshir Stones Information: Lower Lincolnshire Limestone. English Heritage Atlas Area Northamptonshire County Council Date compiled: 02-Nov-10 https://www.bgs.ac.uk/mineralsuk/buildingStones/StrategicStoneStudy/EH_atlases.html

Laycock, E & Yates, T (2000). Observed differences in freeze-thaw performance generated in different brick/mortar combinations. Masonry International, 14 (2) 85-90

Laycock E A 2002 Ten Years of Frost Testing at Sheffield Hallam University. Construction and Building Materials Volume 16, Issue 4 , June 2002, Pages 195-205

Morell, B. 2013 Collyweston Slate Trials [unpublished report] Report date 25th April 2013

Richardson, Linsdall 1929 The country around Moreton in Marsh Sheet 217. Memoirs of the Geological Survey. H.M.S.O: London

Purcell D, 1967. Cambridge Stone. Faber and Faber Ltd, London. Pp 59-63

Salzman L F, 1952. Building in England down to 1540. A documentary history. Oxford University Press, Oxford. p 232.

Selwood, B.W & Valdes, P.J. (2008) Jurassic Climates. Proceedings of the Geologists' Association, 119,5-17

Woodward, H. B 894 1894 The Jurassic Rocks of Britain, vol IV The Lower Oolitic Rocks, Geological Survey of Great Britain London: H.M.S.O, 1894 pp170, 182, 194, 197, 482-484

Worden R & Morad S (Eds) 2009 Clay Mineral Cements in Sandstones (Special Publication 34 of the IAS) ISBN: 978-1-4443-0434-3 Wiley-Blackwell

Acknowledgements

The authors would like to acknowledge the following organisations for their part in the work

- Burghley Estate
- Historic England (formerly English Heritage)
- Messenger Construction Ltd.
- Totally Stoned Company