ALVEOLAR EROSION AND ITS CONSERVATION RECOMMENDATIONS FOR THE SANDSTONE MASONRY AT DURHAM CASTLE

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A THESIS

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to my family...
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Chapter 1: Introduction

Historic Background

Durham Castle is a Norman fortress located at Durham, in the north of England. [Figure 1.01] Construction of the castle began in 1072 under the direction of King William the Conqueror as a border fortress against northern England and Scotland. Following King William’s commission of the building, it was transferred to Walcher, the Bishop of Durham. Since that time the castle has remained in the possession of the prince-bishops of Durham for 750 years. Each bishop has subsequently made changes to the castle including additions and restorations. In 1837, the castle was donated by Bishop Van Mildert to the University of Durham and still houses it to the present day.¹

Durham Castle was built in the traditional Norman Motte and Bailey form that is still visible today despite the many changes throughout its long history. Subsequent additions have integrated the various architectural styles throughout the structure. [Figures 1.02 - 1.03]

In 1986, Durham Castle was designated by UNESCO as a World Heritage Site (WHS), along with Durham Cathedral and the site known as Palace Green; its significance based on its architectural features, its political, social, and religious history reflecting the power of the Norman
conquerors and Prince-Bishops, its significant role in the creation of the town and community, its role as a home to the University of Durham, and its collection of heritage skills.\textsuperscript{2,3}
figure 1.02. durham: the castle courtyard. university of durham archives. 1900.

figure 1.03. durham: the castle courtyard. tiffani simple. 2009.
The main goal of the World Heritage Site (WHS) Management Plan is ‘to describe an approach to the future management of the Durham Cathedral and Castle WHS that will retain and strengthen the balance between conserving the historic environment, enhancing the local character and amenities of the WHS and surroundings with the need to keep the WHS in active and appropriate use, primarily as a religious, educational and residential working environment’.4 [Figure 1.04]
thesis focus

This thesis studies the sandstone masonry of the castle with a focus on its current condition, deterioration mechanisms, in particular, alveolar erosion, and the performance of previous repairs. This information will then be used to test and evaluate the current stone and recommend treatment methods in addition to the current stone replacement.

The current key issue of stone deterioration was identified in the ICOMOS-UK 1995 Monitoring Review of UK World Heritage Sites. The castle was placed on English Heritage’s Buildings at Risk register and categorized as Grade C – ‘suffering slow decay; no solution agreed’. Currently, this issue has still not been resolved.

The exterior stonework of the castle has been deteriorating at least since the earliest photographic records dating to the late 19th century. The stonework is of a local sandstone laid in both plain and carved ashlar blocks. According to Maurice Tucker, there is variation in the stone – from fine to medium grained with a few coarse grained stones, well laminated to thick bedded, from cross-laminated to cross-bedded, and from pale to darker brown. The upper Carboniferous sandstones used for the castle were deposited in a river delta environment, with the sediment derived from the north/northeast. The sandstone is mostly a lithic quartz arenite. Some sandstone is more clay-rich; the quartz
is mostly monocrystalline with some polycrystalline quartz. There are also micas present, mostly muscovite with a little biotite. The cement is constituted by clay minerals, with some quartz overgrowths. The brown color is due to varying amounts of limonite/goethite. This iron oxide is mostly derived from the breakdown of clay and mafic minerals, and possibly pyrite.5

The original (historic) stone and its weathering found at the castle appear to be consistent across the entire site, regardless of age or location and are representative of other buildings in the area, such as Durham Cathedral. The obvious diagnosis therefore appears to be related to the geo-chemical nature of the stone more than any other single factor. Alveolar erosion is particularly evident and poses the greatest risk given its resultant loss of stone and unit volume, leading to visual disfigurement and structural instability.

Because of this long-lived problem, composite mortar ("plastic") repairs and stone replacement have frequently been performed on the castle. Although there is a long history of repairs to the stonework, hazardous structural conditions in the early 20th century forced into action a massive restoration project to save the castle. In 1927, Oscar Faber, a consulting engineer, prepared a structural assessment and report on the castle and cathedral that led to one of the most ambitious restoration projects in England before the Second World War.6
Between 1929 and 1939, an extensive restoration campaign was implemented on the courtyard of the castle to prevent the building from collapsing into the river. During this restoration, Faber underpinned the building with brick and concrete to reduce the pressure on the two unstable walls. Cement was also used to strengthen the walls themselves and a “synthetic stone paste” was used for mortar repairs on the exterior stonework. The joints were also repointed along the exterior walls of the courtyard during the 1930s restoration.

More recently, the primary conservation method in use on the castle’s stonework is total replacement in kind of the stone. Once the stone has deteriorated to a certain depth, generally from alveolar erosion, the entire block is replaced with a local sandstone. This research seeks to expand the treatment options by studying the composition of the stone and its deterioration mechanisms and patterns and recommending compatible methods of consolidation alone and in association with composite repair as a means of avoiding eventual large scale loss and replacement of unit masonry.
Summary background research was first performed on Durham Castle including its use, construction and restoration history. Following this, a technical literature review was compiled on alveolar erosion including its various origins and enabling factors. Investigation was then carried out on the composition of the stone including its mineralogy and microstructure, to better understand its weathering and deterioration tendencies. This informed a reading of the conditions in general on the castle, and in particular, for the elevation recorded in detail during summer 2009. Additional factors to be considered included the environment and microclimate, construction and detailing, and previous alterations, treatments, and maintenance regimes. After this was completed, material analysis and physical and mechanical testing were completed on bulk samples taken. [Appendix A – Methodology]

Site research began in summer 2009 using the University of Durham’s archive collection. A mapping and chronology was developed to better understand the construction, alterations, maintenance and conservation that have taken place on the structure over time. Along with understanding the castle’s history, it is imperative to study the stone’s current condition. Using a representative elevation of the castle, a graphic survey was conducted at the time to record the many stone conditions present. [Figure 1.05]
figure 1.05. Durham castle plan with pilot elevation highlighted. Altered plan based upon drawings by Smiths Gore surveyors. 2010.

figure 1.06. Capturing elevation photos for rectification. Tiffani Simple. 2009.
A detailed ortho-rectified photographic elevation of the east façade of the main courtyard was constructed and the condition of every stone on the east façade was documented. [Figure 1.06-1.08] Various conditions were identified based upon the ICOMOS ISC Stone Condition Glossary and all of the conditions present on the façade were recorded graphically in AutoCAD and transferred into GIS using ArchView. Scaled photographs of all of the conditions were also taken to represent the range of severity of each condition. Using this glossary as a guide, the condition of each stone on the east elevation was documented and recorded, including construction evidence, alterations, and deterioration conditions. Wherever possible, past conditions were annotated using historic photographs of the elevation.
laboratory analysis and performance testing

After the documentation was completed of the elevation’s conditions, stone, mortar and previous patching samples were collected for further analysis and testing at the University of Pennsylvania’s Architectural Conservation Laboratory (ACL). [Figure 1.09] Material analysis and physical and mechanical tests were conducted including thin section petrography, scanning electron microscopy, moisture and soluble salt identification, clay identification using x-ray diffraction, gravimetric analysis, porosity/permeability, water absorption/desorption, and compression strength testing. [Figure 1.10]
figure 1.09. cutting durham stone samples in the university of pennsylvania fabrication lab. tiffani simple. 2010.

figure 1.10. performing sem analysis at the regional nanotechnology facility at the university of pennsylvania with lolita rotkina and dr. charola. tiffani simple. 2010.
Following the initial material analysis and physical and mechanical testing, treatments were recommended for application and analysis to determine the effectiveness of these treatments in reducing surface deterioration of the sandstone. Consolidation will also be evaluated in association with its compatibility with composite repair. These treatments will then be applied in situ in a pilot test area on the same east façade of the courtyard and monitored for their effectiveness.

The ultimate goal of this thesis is to analyze the current stone and propose additional conservation treatments at Durham Castle to maximize the retention of original stone through both preventive and remedial treatments before replacement becomes necessary. It seeks to extend, not replace, the current program of stone replacement and conservation.

5  Tucker, M.E., e-mail message to author, October 28, 2009.
6  Faber, Oscar. “The underpinning of Durham Castle”.
Chapter 2 : Brief Historical Background of Durham Castle

construction evolution

In the early 11th century, the Normans invaded and conquered England replacing the Saxons as the ruling class. These battles raged throughout the country, including the northern area surrounding Durham. The new Norman king of England, William, entrusted the region to the Earl of Northumbria; however rebellion continued and several earls were murdered within three years. Finally in 1069 King William traveled to the north himself with an army and placed another earl in control of the area, Waltheof, Earl of Northumbria. In order to keep future earls safe in Durham, William decided to build a fortress in 1072, i.e., Durham Castle.

In 1072 the construction of Durham Castle began under the supervision of Waltheof, until he was murdered in 1075. He was succeeded by Walcher, Bishop of Durham who succeeded in joining the positions of Earldom and Bishopric. Since then, the castle has become the primary residence of the Bishop of Durham and it has remained in the hands of successive bishops for approximately 750 years. Each bishop made additions and alterations to the castle that can be dated back to Walcher’s initial decisions. [Figure 2.01]
Bishops who contributed some of the most significant alterations to Durham Castle are Pudsey, Bek, Hatfield, Langley, Tunstal, Cosin and Chandler. Bishop Walcher, who was the Bishop of Durham from 1075 to 1080, oversaw the initial construction of the fortress begun by Waltheof including the wall, keep and gatehouse. He also added the Great Hall and the Norman Chapel, which is one of the castle's most intimate and significant spaces. When Bishop Flambard came into control in 1099, he built a stronger and higher wall of stone around the peninsula that enclosed the castle's inner bailey and also reconstructed the Gatehouse.
In addition to his reconstruction efforts, Flambard erected the North Hall of the castle. The subsequent Bishop Rufus made no significant alterations to the castle.

After the death of Bishop Geoffrey Rufus in 1140, Durham Castle was seized by William Cumin and was taken from the prince-bishops for thirteen years. In 1153, it returned to the prince-bishops but much of the area was devastated from Cumin’s reign. Bishop Hugh le Puiset (Pudsey), who ruled from 1153-1195, restored much of the damaged areas. However other problems beset the castle including a serious fire that destroyed much of the town and also damaged the castle. Pudsey had the damaged buildings restored that included a significant portion of Flambard’s north range.

Almost a century later, by the late 13th century, another significant addition was made to the interior of the castle. In 1284, Bishop Antony Bek added the Great Hall, which has continuously been the main central space of the castle since its construction. In 1345, Bishop Hatfield replaced Bishop Bek and had the Great Hall extended adding large windows to it, seemingly enlarging the space. Hatfield also rebuilt the keep. It was enlarged into an octagonal shape and constructed of stone replacing the former timber structure system. Enlarging the keep required enlarging the mound so that it is now adjacent to the Norman Chapel and blocks the windows on the east side. Hatfield also
reconstructed the roofs from a flatter roofline to its current higher pitched open timber roof.⁶

During the reign of Bishop Thomas Langley from 1406 to 1437, the last significant addition to strengthen the fortress, the rebuilding of the North Gate, was completed. Although the castle was initially built as a fortress, its primary purpose was to serve as a home for the prince-bishops. In 1500, Bishop Richard Fox added the medieval kitchens, which are still in place adjacent to the Great Hall. Fox also shortened the Great Hall during his time and added four floors of rooms at the south end. After Bishop Fox, no significant alterations were made until Bishop Cuthbert Tunstal in 1530. Tunstal had an additional chapel, Tunstal’s Chapel, added. Tunstal’s Chapel was built in the north-east corner of the courtyard with a stairway that gave access to the chapel from the courtyard. Tunstal also altered the Gatehouse, making the entrance wider.⁷

In 1642, the Civil War between Crown and Parliament began and the castle suffered from neglect. The Bishopric was abolished and church land was sold off. During this time, the castle was used as a hospital to house many suffering from dysentery. In 1660, when the Bishopric was restored, the castle returned into the hands of the bishops. Bishop John Cosin was the first bishop after the return finding the castle uninhabitable, major restorations were conducted under his rule. These
are the most extensive alterations to date.

Bishop Cosin’s alterations accentuated the castle’s purpose as a bishop’s home rather than as a fortress. He demolished the barbican and filled in the dry moat; replacing them with a driveway flanked by gardens which are now known as the Fellows’ and Master’s gardens. The keep’s mound was also landscaped with three terrace walks. Cosin also constructed a stair tower in the north-west corner of the courtyard. This staircase is known as the Black Stairs because of its rich ebony color. Also among Cosin’s design decisions was the addition of a porch at the grand entrance of the castle and the refurbishment of Tunstal’s Chapel. Because of continuing stone deterioration that affected its structural stability, the building in the south-west corner of the courtyard was refaced. Both the buttresses along the Hall’s east wall and the walls of the keep mound were strengthened as well. In 1672, Bishop Cosin passed away before his alterations were completed. The next bishop, Bishop Crewe, continued the alterations begun by Cosin and he also began repair work on the keep in 1714. This was continued by Bishop Thomas Thurlow who had the upper stories of the keep demolished in 1789.

By the middle of the 18th century, Bishop Chandler did some alterations, mostly on the exterior of Puiset’s Hall. The north wall was partially rebuilt and the south wall needed substantial repairs. As it was leaning
outwards, it needed to be cut back and refaced. Large beams above the ceiling and below the floor of the Hall were also placed to stop any further movement in the walls.

Bishop van Mildert (1826 to 1836) was the last crowned prince-bishop of Durham Castle. During his time the controversy over the power of the church began and after van Mildert’s death the remaining powers were passed to the crown. Durham Castle was then transferred to house the newly established University of Durham and the keep was once again reconstructed in 1840. Although it was the third time that the keep was reconstructed, it still includes a portion of the original stonework. During the transition to University, the castle hall was also altered to include the installation of stained glass in the north window of the hall.

To date, the castle still retains its original Norman Motte and Bailey form. Its original military intentions are reflected in its position on the northern portion of the peninsula in Durham, 35 meters above the River Wear. These are also emphasized in the walls surrounding the castle, the shape of the courtyard, the gatehouse used to enter the courtyard and the keep placed on a mound overlooking the peninsula and outer city. [Appendix B – Castle Drawings]
Deterioration

Durham Castle sits on layers of clay, shale and broken freestone. This has caused problems for the castle structure since its original building. From the earliest recording in the 12th century, the North Hall began to subside and the north-west tower was constructed to stop this movement in the 13th century. Although this solution worked for some time, by the mid-18th century the north and south walls shifted apart, threatening to collapse the north range again. As previously discussed, this was altered in Bishop Chandler’s time by cutting back and refacing the walls as well as inserting wooden beams.

By the early 20th century, the North Hall was once again in poor condition and the walls continued to move outward. Metal tie-rods were then inserted into the structure to prevent the outer walls from moving further. These structural conditions continued to worsen as previous repairs did not address the causes but rather symptoms, sometimes creating an even larger problem. It was discovered in the 1920s that the metal tie-rods intended to stop the south wall from slipping, by tying it to the north wall, failed. Instead of making the south wall stable, the south wall continued to slip and then began to drag the north wall with it. Because of the continuous deterioration of the castle, a major restoration campaign was begun to address these structural problems in the 1930s.
Apart of the structural problems, the most important deterioration problem associated with Durham Castle is the erosion of its stonework. [Figure 2.02] The castle is constructed in ashlar blocks of a local sandstone. This sandstone is readily available and can be seen in numerous buildings throughout Durham as well as in the Cathedral. The weathering of the stone can be observed not only in the castle, but in other buildings as well. Therefore, it appears that the deterioration is associated with the geo-chemical nature of the stone, regardless
of its age or location. The sandstone is poorly indurated and erodes differentially over time. [Figures 2.03-2.05] Although it is primarily a surface phenomenon, given enough time, the deterioration will result in large cavity losses known as alveolar erosion. This appears as cavities of variable shape and size on the stone surfaces13 leading to a significant amount of stone loss, thus increasing the risk for structural instability.

figure 2.03. note the advanced deterioration of the portal columns. durham university class photo at east facade entrance. university of durham archives. 1904.
figure 2.04. note the variations between figure 2.3 and 2.4 deterioration over a six-year time span. durham university hockey team photo at east facade entrance. university of durham archives. 1910.
Figure 2.05 note the variations between figure 2.3, 2.4 and 2.5 deterioration over a fourteen-year time span. Durham University Society photo at east facade entrance. University of Durham archives. 1919.
past treatments

Because of the long-lived problem of stone deterioration at the castle, numerous repairs and treatments have been made over time. Historically, once the stone began to erode, the weather-eroded surface was chiseled off to obtain a flat surface again.\(^{14}\) This method was eventually outdated by re-facing the eroded stonework and bringing it back out to its original face plane. From the initial construction of the castle to the early 20th century, repairs and alterations were only performed when masonry had become structurally unstable or had deteriorated to a significant extent. Even then, only specific portions were repaired. It was not until the early 20th century that the castle was in such a serious state that a major restoration campaign was implemented.

In 1927, Oscar Faber, a consulting engineer, prepared a structural assessment report on the castle and cathedral that led to one of the most ambitious restoration projects in England before the Second World War. Faber stated in his Report on the Condition of the Fabric that the castle was “in a highly precarious condition and any delay in dealing with it may result in total or partial collapse”.\(^{15}\) Soon thereafter a funding campaign was started to finance the restoration. [Figure 2.06]
To address the structural problems that had long occurred on the south wall and then more recently on the north wall, Faber suggested underpinning the building with brick and concrete; thus reducing the pressure on the two walls. The west range also needed stabilizing from the pressure of the building on the bedrock, since it had caused the west wall to start to move outwards and downwards. To remedy this, Faber had the walls strengthened by drilling holes through them and inserting cement. Steel tie-rods were then inserted to anchor the western retaining wall and the west wall to blocks of concrete under the courtyard. [Figures 2.07]
Since the exterior stonework was so badly eroded, much of the stonework required patching or full replacement. Synthetic stone treatments, i.e., mortar “plastic” repairs, were performed across the entire façade where necessary. [Figure 2.08-2.09] During this restoration campaign, cement-based mortar was often used for patching along with other treatments. Although the patchwork of mortar repairs stands out against the less eroded stone, it does not appear to be increasing the erosion of the stonework. Along with the stonework, there was also a significant amount of mortar loss. This required the repointing of all the facades facing the courtyard. [Figure 2.10] To replace the lime mortar, a harder,
figure 2.08. synthetic stone treatment during the restoration campaign. university of durham archives. 1936.
figure 2.09. synthetic stone treatment during the restoration campaign. university of durham archives. 1936.

figure 2.10. removing mortar for repointing during the restoration campaign. university of durham archives. 1936.
denser cementitious mortar with large aggregate was used. This mortar is still in place today and is rarely missing except in places where it has been chiseled out to replace stonework.

Historically, if the stone was significantly eroded, the decision was made to completely replace it with a similar stone. This method is still the predominant one today due to the availability of local stone and of traditional skilled labor force. For the past fifty years Black Pasture Sandstone and Dunhouse Stone have been used for repairs and restoration at Durham.\textsuperscript{19} Black Pasture Stone is obtained from quarries located near Chollerford, Northumberland\textsuperscript{20} and Dunhouse Stone is quarried near Darlington.\textsuperscript{21} These two sandstones are from the same region as the sandstone that had been used in Durham Castle since its construction. They are very similar in composition and therefore work well for replacement. Although when installed it has a similar appearance to the patching, the replaced stone weathers quickly and soon looks much like the rest of the stonework in color.

Faber in addition commented on the damaged roof systems of the castle that have also been a continuous problem. Following repairs to the roof in the 1930s, additional restoration work was needed in the 1990s to address dry rot of the timbers and leakage in the roof space.\textsuperscript{22} Much of the roof has traditionally been covered in metal, usually lead, and external failure and internal condensation have led to a lot of the decay
in the supporting timber structure requiring continuous inspections and repair.\textsuperscript{23} The last large restoration project from 2004 to 2006, included the repair and replacement of most of the castle’s roofs.\textsuperscript{24}

Currently, replacement of the deteriorated stone is the only masonry conservation effort in effect at the castle. Stone masons work specifically at Durham Castle to prepare stone for replacement. While this method has been the standard approach for conservation at the castle for hundreds of years, recurring replacement will eventually lead to nearly wholesale replacement of original fabric. Analyzing and developing other conservation methods is not only beneficial to the conservation of Durham Castle but imperative for the environment as well.

\textsuperscript{1} General Note: The information in this section is based primarily upon Richard Brickstock’s book Durham Castle: Fortress, Palace, College.


\textsuperscript{7} Brickstock, Richard. Durham Castle: Fortress, Palace, College. p. 35.


\textsuperscript{9} Brickstock, Richard. Durham Castle: Fortress, Palace, College. p. 38


\textsuperscript{13} ISCS Stone Glossary.


Alveolar erosion, the formation of cavities of variable shapes and sizes on the stone surface, is a condition that occurs frequently in the sandstone of Durham Castle. It has also been referred to as honeycomb weathering, tafoni, stone lattice,\(^1\) and cavernous weathering.\(^2\) This type of weathering has a very distinctive pattern. Starting as a surface phenomenon, once a sufficient amount of stone has weathered, i.e., both regarding surface extent and depth within the alveolus, it begins to have structural implications. Although the erosion patterns are visually apparent, the surface appearance does not give enough information to explain the changes that occur in the stone during this weathering.\(^3\) Although this phenomenon was already described in the literature from the 1920s, the discussions on the causes of this weathering continue, and there are multiple factors involved in it.

Alveolar erosion is found in various rock types, Mustoe listed several occurrences referenced by other investigators and found that most incidents occur in homogeneous rocks such as sandstone, limestone and granite.\(^4\) It has been found in sandstone in southern France, Spain, Italy, Australia, New South Wales, New Zealand, New Mexico, Colorado, Arkansas and Wisconsin. In limestones it has been reported in France,
Egypt, and Morocco, while in granite and other rock types, it has been found in Antarctica, California, Hawaii, Western Australia, Finland and the Alps. Many occurrences of alveolar erosion have been found in coastal areas and canyons although it develops in various other locations across the world.⁵

It is known that in areas where alveolar erosion begins, the rock surface has a lower strength.⁶ Motterhead, using a strength measuring machine known as a Cone Intender, found that the strength of honeycombed sandstone could be reduced to 77% of the strength of the un-weathered portions of stone.⁷ Many studies have been made to identify the factors that lead to alveolar erosion and to determine why certain portions of stone weather preferentially over others. This has resulted in various hypotheses. Because of the variety in occurrences of this deterioration pattern, i.e., type of rock, location and environment, it may be assumed that not necessarily the same agents are the cause. Specific causative agents have been identified in the many case studies examined, yet each site is different depending upon the factors involved at that site.

possible causes/influences – water, wind, salts, clays

Alveolization has been known to result from a combination of both physical and chemical weathering. However, it appears to be more of a physical phenomenon than a chemical one.⁸ ⁹ G.E. Mustoe in
The Origin of Honeycomb Weathering stated that although the origin remains a controversy, microscopic examination of the weathered surfaces shows that alveolar erosion results from the disaggregation of mineral grains rather than from chemical decomposition. E.M. Winkler also acknowledged that alveolar erosion evolved from mechanical disaggregation with a combination of surface hardening.

Mustoe concluded from his research that in most cases, well-developed cavities tend to form along bedding, joint planes, or other areas of compositional weakness. The specific cause of alveolar erosion is debated but several external agents have been suggested such as wind erosion, salt weathering, frost shattering, and exfoliation. While these explanations of the cause of alveolar erosion have been postulated, it is the combination of several factors acting together that result in the phenomenon.

In a study conducted by Charola at Cerro Colorado, Argentina, stone deterioration patterns were attributed to the internal structure of the stone. Through observation and SEM analysis, it was determined that the porosity differences in the stone were causing redeposition of the binding material resulting in strengthening and weakening parts of the stone, in response to water and wind weathering.

Salt weathering is one of the most prominent explanations of alveolar
erosion. This was first expressed by Hume who concluded that alveolization resulted from the physical action of salt crystallization.\textsuperscript{17} Winkler found that this was often controlled by the presence of salts and by the fabric and structure of the stone.\textsuperscript{18} The presence of salts is not required for alveolar erosion to form; however they have been known to accelerate the process.\textsuperscript{19} Young stated that in coastal and arid environments, salt crystallization is a prominent mechanism in the formation of alveolar erosion and in more humid environments, the hydration of clays acts as an alternative mechanism for forcing apart grains within the rock and thus causing cavernous weathering.\textsuperscript{20}

Young’s study of the cavernous formations in coastal locations of Australia, pertaining to salt as an agent in the development of such weathering, discusses salt crystallization forces and its disintegration of rock. After examining salts in study areas, Young found that salt crystals were not infilling voids as suspected, but that they were scattered in samples, therefore enhancing the dissolution of silica but not forcing apart grains by exertion.\textsuperscript{21} As these grain-to-grain quartz contacts and cementing clays are dissolved in the stone, the weathering proceeds. These grains are then left separated and the voids become widened by dissolution of silica.\textsuperscript{22} Young found that soluble salts in water percolating through quartzose sandstone deeply influence the promotion of granular disintegration. The salts do not force apart grains by crystallization, rather, they increase the rate of silica solution of the
quartz grains and thus promote weathering.\textsuperscript{23}

Stone containing clays has also been noted as being more susceptible to alveolar erosion. Cavernous forms in over 20 locations across Finland were studied by Kejonen and co-workers and found to contain clays such as illite, kaolinite and vermiculite as well as mixed-layer clay minerals.\textsuperscript{24} These formations developed in a variety of rock types including gneisses, granitoids, amphibolites and breccias.\textsuperscript{25} Weathering processes responsible for alveolar erosion in Finland were flaking and granular disintegration. Granular disintegration is a physico-chemical weathering that attacks the weak parts of the rock generating clays. These, through their expansion-contraction cycling, forces apart the stone grains increasing the voids within it. Typical granular disintegration weathering resulted in alveolar erosion as found in various locations across Finland.\textsuperscript{26}

\textit{formation of thin walls – variation in strong/weak areas, internal stone structure}

While there are explanations of the weathering of the stone leading to alveolar erosion, these do not explain the formation of the thin walls that remain between the cavities. Early investigations suggested that ions released by weathering within the cavities precipitated on surrounded surfaces, causing the walls to become strengthened. This
hypothesis was referred to as “case hardening”. Other explanations for the hardened walls is the dissolution of iron minerals, stone porosity causing preferential redeposition of the binding material, and the deposition of silica beneath the stone surface forming a hard layer.

Mustoe concluded from his case study of Chuckanut formations along the Puget Sound that hardened layers resulted from the dissolution of iron minerals. He found that where the hardened layers were well-developed a dissolution of iron minerals occurred within the arkose. The durability of the rock in these hardened layers resulted from the redistribution of iron and aluminum content, however not from a high degree of accumulation. Charola and co-workers found that the hard areas of alveolar erosion were formed where the less soluble material, silica or iron oxides were deposited and the weak areas were a result of the binding material dissolving or where soluble salts were enriched.

process of formation

Winkler discussed the agents involved in the formation of alveolar erosion and suggested the following hypothesis. First, some stone porosity is essential to allow moisture to travel through the stone. Second, dissolved calcite and silica, from the calcitic binder and quartz and feldspars, respectively, in the sandstone move to the stone surface. The deposition of irregular layers of silica beneath the stone surface
can form a hard layer while the calcite, goes into solution moving into the porous sandstone and to the stone surface. The uneven redeposition near the surface is what results in an uneven weathered surface, developing into honeycombs. Third, water-soluble salts cause mechanical disaggregation in protected areas. Finally, the introduction of calcite or gypsum from mortar, cement or concrete into porous sandstone provides material for a partial shield. Therefore, cavities begin to form by the dissolution and differential uneven case hardening in porous rocks. Small cavities then start to form along thinly hardened parts of the shield and the presence of salts enhances the crumbling inside of the cavities.

For the Cerro Colorado, Córdoba, Argentina case study, Charola concluded that alveolar erosion resulted from a combination of a redistribution of the binding material within the stone forming alternating indurated and weakened zones, strong winds producing rapid evaporation and the presence of fairly soluble salts.

Pauly was one of the first to investigate alveolar erosion and conditions required for its formation and evolution. Pauly studied the material and chemical composition of stones from seven different sites in France all of which showed alveolar deterioration. From his laboratory analysis Pauly found alveolar erosion is based primarily upon hygroscopic salts and variations of moisture transport that is affected by rain and
wind. In Pauly’s laboratory simulations, cavities formed differently depending upon hygrometrical conditions. Pauly concluded that alveolar deterioration is due to the action of soluble and hygroscopic salts that are capable of interacting quickly with changes of relative humidity.

Pauly concluded that the context of the building has just as much affect on the process of alveolar erosion as the properties of the stone itself. So while soluble salts play a large role in the formation of alveolar erosion, the more hygroscopic salts play an even more significant role as they are enabling factors of deterioration in conjunction with wind, rain, and relative humidity.

rate of formation

The time required for the formation of alveolar erosion has not been determined. A rate of development was discussed by Tsuguo Sunamura who compared alveolar erosion development to an exponential growth rate and not a linear one as first expected. It was initially thought that as cavities begin to form, they will continue to erode at the same rate becoming larger and deeper. However, once alveolar erosion begins to form, it will continue at a faster rate. At first, a certain amount of time is required for the rock surface to deteriorate and to weaken prior
to alveolar erosion initiation. Then once alveolar erosion has begun forming and cavities increase, they will form at an exponential growth rate.

At Durham Castle, archival photographs provide an approximate rate of formation of alveolar erosion. The first class photo from 1898 shows some deterioration of the stonework, before cavities began to form in most the stone blocks and only slight indentations as are seen on other blocks. [Figure 3.01] Only 5 years later, the class photo from 1903,
the stone has cavities formed in several blocks and significantly larger cavities in other stones. [Figure 3.02] Although these photos do not show the rate of stone weathering before alveolar erosion begins to emerge, it shows an increase in the deterioration rate that suggests a confirmation of Sunamura’s exponential hypothesis, since after the cavities began to form it only took 5 years to show a significant increase in deterioration.

Figure 3.02. University of Durham class photo, University of Durham archives. 1903.
7 Ibid. p. 742.
9 Young, A.R.M. Salt as an Agent in the Development of Cavernous Weathering. p. 963.
13 Ibid. p. 108.
14 Ibid. p. 112.
16 Ibid. p. 322, 324.
17 Hume, W.F. Surface Features.
19 Ibid. p. 176.
20 Young, A.R.M. Salt as an Agent in the Development of Cavernous Weathering. p. 963.
21 Ibid. p. 964-5.
22 Ibid. p. 965.
23 Ibid. p. 966.
25 Ibid. p. 315.
26 Ibid. p. 318.
28 Ibid. p. 113.
29 Charola, A.E. Preliminary Studies of Deterioration Patterns at Cerro Colorado, Argentina: Open Cave Formation and Honeycomb Weathering. p. 324.
34 Ibid. p. 177.
37 Ibid. p. 78.
38 Ibid. p. 79.
40 Ibid. p. 746.
Chapter 4 : Pathology

site description

Durham Castle sits on the northern edge of the Durham peninsula, surrounded by the River Wear which is one of three main rivers that pass through the city. [Figure 4.01] Durham Castle was built on a load of glacial drift up to 5m thick layered on soft shales and sandstones with thick seams of coal scattered throughout.¹ [Figures 4.02-4.03] These

figure 4.01. durham aerial view. google earth. 2009.
Carboniferous rocks that have provided the foundation for the Castle have proved to be weak and had to be underpinned in the restoration campaign of the 1930s. Concrete tunnels and ties were driven into the bedrock to stabilize the castle as well as the entire Wear bank that supports it.\textsuperscript{2}
Figure 4.03. Geological map of Durham County. http://www.soton.ac.uk/~imw/Geology-Britain.htm 2008.
Durham’s location in northeast England and its proximity to the North Sea, exposes it to colder temperatures than the southern and western parts of the United Kingdom. Durham has a temperate climate like most of the United Kingdom with its coldest temperatures in January and it’s warmest in July. According to the latest Census from 1971-2000, temperatures range from 0.6°C to 19.8°C with a maximum average of 12.5°C and a minimum average of 5.2°C.³ [Table 4.01]

Durham encounters approximately 645 mm of rain each year⁴, which significantly affects the stone conditions observed at the Castle. With the high amount of rain and cooler temperatures, Durham is at risk of many freeze/thaw cycles; some 29 per year.⁵ Freeze/thaw cycles occur when the stones are wet and when the air temperature drops below freezing and then returns above freezing without time for the moisture to evaporate. Freeze/thaw cycling has adverse effects on buildings especially when a high amount of precipitation is involved and when the stone is susceptible to moisture-related decay.

The wind speed and direction may also have an effect on the weathering patterns at Durham. On average, the annual wind speed at Durham is 7.5 mph with a prevailing direction that ranges between the south and southwest.⁶ However, whirlwinds may develop within the courtyard.
Table 4.01. Durham climate data.

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existing site conditions and analysis

A conditions survey was conducted on a representative elevation of the courtyard of Durham Castle during the summer of 2009. Based upon the ICOMOS ISC Stone Condition Glossary, all of the conditions were defined and recorded using AutoCAD and ArcGIS with a total of 26 different conditions identified on the Castle. [Appendix C + D] These various conditions include repairs, alterations and deterioration. The 26 conditions are:

Total repair is the complete replacement of a stone unit. When stone has deteriorated to a certain extent, the unit is removed and replaced with a new sandstone ashlar block. Total repair occurs extensively at Durham Castle and is currently the most used method of repair. From the elevation surveyed, 34% is total repair. [Appendix C.01, D.01]

Partial repair is the partial replacement of stone in kind (i.e. Dutchmen). This repair method occurs when a portion of the stone has deteriorated and only a segment of the unit needs to be replaced. Partial repair is not as frequent at Durham, but still has occurred in specific areas. [Appendix C.02, D.02]

Patching refers to stone surface replacement with mortar. Patching appears to have been conducted on stone that displays surface loss and
is associated almost exclusively with the 1930s restoration campaign. The stone is cut back to reach solid surface and then infilled with mortar, instead of refacing it or replacing the entire stone. Since alveolization and other surface deterioration problems occur extensively at Durham Castle, patching is often the first means of repair before an entire stone is replaced. It is therefore seen across the entire elevation and approximately 45% is patching repair. [Appendix C.03, D.03] Total repair (partial and complete) recorded of the studied elevation suggests that that only 21% of the original stone surface remains.

Metallic elements noted on the elevation include all iron and copper alloy insertions that are embedded into the masonry wall. Metallic elements are present on the elevation towards the bottom of the wall to secure wires that were run on the exterior of the building and for other methods of support and fastening. [Appendix C.04, D.04] These can cause associated conditions such as cracking and disfiguring stains due to corrosion.

Alveolization is the formation of cavities of variable shapes and sizes on the stone surface. Alveolization is one of the most serious conditions observed on the structure and was noted on approximately 4% of the elevation. [Appendix C.05, D.05] Presumably the presence of excessive alveolization was addressed by partial and total repair some time in the past.
Cracking was noted in both minor and major categories. Minor cracking refers to fractures varying in orientation and depth, and less than 1/8” in width. Minor cracking is usually seen in isolated cases along the bedding mortar and stone interface and then often extending through stone in specific sections. Major cracking refers to fractures that are greater than 1/8” in width and usually have structural implications. Major cracking does not occur as frequently as minor cracking, and is observed on buttresses and other locations with high structural loading. [Appendix C.06, D.06 + C.07, D.07]

Peeling is the thin uniform delamination of tooled and patched surfaces. Peeling can be partial where a portion of surface still remains attached (Detached Peeling) or it can result in a total loss stone surface (Gone Peeling). Peeling is seen in Durham Castle in many places where total repair has occurred, mostly on windows across the façade suggesting the condition is associated with the replacement stone. Approximately 1% of the surface displays Detached Peeling and 2% Gone Peeling. [Appendix C.08, D.08 + C.09, D.09]

Friability is the disaggregation of stone surface usually in association with moderate and major loss. Friability generally begins at the stone surface continuing to various depths and results in smoothed surfaces or those that appear sugary or powdery. 25% [Appendix C.10, D.10]
Visible Detachment is the physical separation of the stone into layers often accompanied by partial loss. Visible detachment is observed on approximately 12% of the elevation. Blind Detachment is not visible but can be detected by tapping the stone to determine voids or discontinuities beneath the surface. Blind detachment covers 1%. [Appendix C.11, D.11 + C.12, D.12]

Linear Erosion appears as a striated loss of stone surface and is related to the sedimentary bedding and the orientation of the sandstone units where the bedding is perpendicular to the surface (i.e. natural bedded). Linear erosion can sometimes create cavities similar to alveolar erosion along the beds and other times loss appearing in striations. Linear Erosion covers 12% of the elevation [Appendix C.13, D.13]

Moderate and Major Loss occur in stone by various mechanisms. Moderate loss refers to surface loss of less than ½" in depth and major loss refers to surface loss of more than ½" in depth. Loss is usually observed in conjunction with other conditions that have caused the stone to deteriorate. Moderate loss is observed on approximately 4% of the elevation and major loss occurs on 1%. [Appendix C.14, D.14 + C.15, D.15]
Contour scaling is the detachment of stone in stacked layers resembling topography, often in association with visible and blind detachment and is associated with face-bedding installation of the stone (parallel to the surface). [Appendix A.16, B.16]

Flaking is another form of detachment that results in small, thin flat or curved scales on the surface. Flaking occurs often across the top of the elevation and also in the buttress stone. This form of detachment occurs on 1% of the stone evaluated. [Appendix C.17, D.17]

Displacement is the movement of a stone unit from its original position. Displacement refers to any movement out of plane or alignment. [Appendix C.18, D.18]

Efflorescence corresponds to the formation of whitish, powdery crystals of soluble salts on the stone surface. Efflorescence was only noted on a few localized areas across the wall. [Appendix C.19, D.19]

Spalling is irregular loss of stone usually caused by impact or corrosion. Incipient Spalling refers to cases where the irregular fragments are still partially attached. [Appendix C.20, D.20]

Missing Joints are missing or damaged joints, which usually refers to
lost mortar in the elevation. [Appendix C.21, D.21]

Interface Erosion is loss occurring at the interface of original stone and stone repair. Locating interface erosion was significant in determining if repairs were causing or accelerating existing stone deterioration. Interface erosion was only noted on .05% of the elevation surveyed. [Appendix C.22, D.22]

Metallic Staining refers to red-brown discoloration related to iron corrosion and is found near metallic elements such as inserts, and wiring present on the building. [Appendix C.23, D.23]

Vegetation is the presence of higher plant forms, including their root remnants. Vegetation is seen on the roof of the entrance portico and rarely in any other places and suggests areas of high water saturation. [Appendix C.24, D.24]

Guano is bird excrement. The only locations where this is found on the elevation are areas of the large windows. [Appendix C.25, D.25]

Micro-flora is the presence of algae and fungi, and including mosses and lichens, identified as localized areas of black, green or brown discoloration. Micro-flora was noted across the entire façade with large
concentrations on the crenellations, the top of the buttresses, the lower wall and along the window sills—all areas of high moisture exposure and saturation. Micro-flora was recorded on 15% of the east wall. [Appendix C.26, D.26]

deterioration mechanisms & assessments

The east wall of Durham Castle is representative of the many conditions observed throughout the complex that can be attributed to multiple factors. A matrix was established to explore the relationship among the recorded building conditions, probable mechanisms of deterioration and the enabling factors on site such as moisture or bedding orientation. Of the many conditions recorded, those associated with forms of stone loss were studied in depth including repairs (evidence of past damage), alveolization, peeling, detachment, friability, linear erosion and micro-flora. [Appendix E]

The east elevation of Durham Castle contains a significant amount of patched and replaced stone, 45% and 34% respectively. The repair methods suggest that the stone had reached such a state of deterioration that it was necessary to either completely replace it or repair its lost surface. Total stone replacement has occurred on portions of the buttresses, in areas surrounding the buttresses, and the in the carved windows and portal beginning as early as the 18th century. The mortar
patching observed across the entire elevation was installed during the 1930s restoration campaign. Stone ashlar that was deteriorated but could still be salvaged was patched. The extent of these repairs suggests that the stone is intrinsically susceptible to deterioration and the type and severity of the different conditions recorded are a function of associated enabling factors such as moisture access (rising or falling damp), exposure, detail designs, and stone bedding orientation. No patterns of stone decay were found in association with patching to suggest that this repair technique has caused or exacerbated earlier conditions suggesting incompatibility of the “plastic” mortar repairs (aside from aesthetics). [Appendix E.01 - Repairs]

Alveolar erosion, the major conditions studied in this thesis, occurs across the east elevation although it is more prominent on the buttresses and beneath the window sills. This type of deterioration occurs block by block; in some cases the cavities encompass the entire surface area of the stone, in others it only occurs in certain portions of the stone. Alveolar erosion, which was discussed in-depth in the previous chapter, can be the result of various deterioration mechanisms which include salt crystallization cycling and expansion and contraction of clays during wet-dry cycling, while silica formation and iron deposition can act as contributing factors. It is the major deterioration type occurring at Durham attributed to the stone’s geo-chemical nature in conjunction with wind and precipitation (rain and melting snow). The observed
concentration of this condition on the buttresses may be due to the fact that these architectural features project beyond the plane of the wall which makes them more susceptible to wetting and drying cycles. It also occurs more heavily under window sills because of high water saturation from sill collection and disposal. [Appendix E.02 - Repairs and Alveolization]

Peeling—the detachment of a thin uniform single layer of stone independent of its bedding—is a particular type of detachment observed mostly on replacement stone. The windows on the south end of the façade have all suffered from peeling, the protruding oriel window being the worst. Peeling occurs on many tooled surfaces, including both vertical and horizontal surfaces. The windows on the north end of the façade along with the portal do not display peeling like the windows on the south end. This could be a result of the use of two different types of sandstone for the replacements. Factors that could cause peeling to occur are soluble mineral transport that enriches and densifies the surface complete with its original tooling which upon weathering displays differential responses than the stone beneath it. Another factor could be the surface finishing methods that result in subsurface micro-cracking which upon weathering cause surface failure. [Appendix E.03 - Repairs and Peeling]
Detachment, contour scaling and flaking are all subtypes of the stone detaching in multiple layers. Detachment occurs across the façade and is a direct result of the geochemical nature of the stone and its reaction to water. However, flaking, another form of detachment, occurs more on the buttresses and lower zones of the elevation. Because the stone is a sedimentary sandstone composed of bedding strata, these forms of detachment reflect the stone’s stratigraphic structure in the presence of varying amounts of moisture. [Appendix E.04 – Repairs and Detachment and Flaking]

Friability occurs generally across the elevation as well. It is stone dependent and does not seem to be related to location on the façade. The disaggregation of stone seems to be one of the typical conditions that can be attributed to the geo-chemical nature of the stone. Factors that would cause this to occur are high porosity of the stone, the quartz grains not being very compacted thus allowing moisture to enter the stone easily. Freeze-thaw flaking, clay swelling and shrinking, and soluble mineral transport and depletion of the exposed zone may all be factors in causing friability. Friability and detachment are seen both separately and as combined conditions on stones. [Appendix E.05 – Repairs and Friability and Detachment]

Linear Erosion is also a distinct deterioration pattern similar to alveolization however its deterioration patterns appear in association
with the exposed bedding planes in the natural bedded sandstone. Again this form of erosion can also be attributed to salt crystallization, freeze/thaw cycling, or the hydration of clays. Linear erosion is observed on the façade in areas that are highly prone to water such as the crenellations, projecting elements such as windows, and the masonry below the window sills. Because of the vulnerability of the bedding planes when exposed on edges, these areas become more susceptible to micro-cracks, allowing water to enter the sandstone. [Appendix E.06 – Repairs and Linear Erosion]

Micro-flora is an indication of excessive moisture saturation of the façade and therefore tends to occur in places of high water contact such as the crenellations at the top of the elevation, window sills, below windows, and the stonework at grade from capillary transport (rising damp). [Appendix E.07 - Repairs and Micro-flora]

From the various conditions observed at Durham Castle, it can be hypothesized that the main cause of deterioration of the stone masonry is the highly vulnerable geo-chemical nature of the sandstone itself, particularly the presence of expansive clays. Alveolar erosion as well as many of the other conditions that occur are enabled by the porosity of the sandstone and its contact with excessive moisture.
It is concluded that the synergies between the geo-chemical nature of the sandstone, the presence of excessive moisture delivered as rain, melting snow and aerosol (fog), and the context of each element (stone environment), all play a major role in determining the variety of conditions that can be observed on the east elevation of Durham Castle. No single element of these three could have resulted in the damaging conditions present. Further analysis and environmental monitoring will confirm or clarify the mechanisms responsible for the stone deterioration observed at Durham Castle and other buildings in the vicinity.

2 Ibid. p. 30.
5 Engineered Weather Data. Courtesy of Michael Henry, P.E.
6 Ibid.
Chapter 5: Stone Analysis

Introduction to Sandstones

Sandstone is a clastic sedimentary rock of sand-sized minerals or rock grains that are mostly composed by quartz with some feldspar minerals. The grains of sandstone are naturally cemented and the cementing material can be quartz, silica, calcite, clays, and iron compounds. Cementation of sandstones is the primary process that determines the porosity of a sandstone. Calcite cementing material often gives a crystalline texture and appearance to the stone and if a substantial amount of calcareous cement is present, the stone is considered a calcareous sandstone. Silica is one of the most common cementing materials and is formed through dissolution or from alteration of the quartz sand. Clay material can also be a cementing material but is not a desirable constituent because of its plate-like constitution that results in its susceptibility to shrinking and swelling in the presence of moisture and from frost susceptibility. Iron compounds are also found frequently as cementing agents and usually consist of iron oxides or oxide hydrates, or ferruginous clays. Sandstones with iron compound cementation are known as ferruginous sandstones. Other types of cementing mediums such as gypsum and dolomite, also can be found in sandstones, which alter the stone’s properties and composition.

Adams et al. describe the classification of sandstones by their
cementation agents and principal matrix components and classify them according to their sedimentation maturity. Sediment maturity refers to the mineralogical and textural level of composition in sandstones. Mineralogically mature sediments are the stones that contain a high proportion of the most chemically stable and physically resistant minerals such as quartz. Sediments considered mineralogically immature contain less stable grains such as feldspars.4

Folk described the maturity of sandstones by categorizing them according to their cementation matrix and the sorting and roundness of the grains. An immature stage consists of sediments that contains more than 5% clay matrix and grains that are poorly-sorted and not well-rounded. Submature stages of sandstone have less that 5% of clay matrix in the sediment with the grains still poorly-sorted and not well-rounded. The mature stage sediment contains little or no clay with well-sorted but not well-rounded grains. Finally, the supermature sediment stage contains no clay and has grains that are well-sorted and well-rounded.5
Durham Castle is constructed out of local sandstone ashlar blocks. The sandstone of the region is a Carboniferous sandstone that can be divided into two major categories: Coal Measures, Millstone Grit and Carboniferous Limestone. Carboniferous Limestone is a misnomer for the stone, however the division is predominately a limestone in the Midlands and southern England, with proportions changing to sandstone towards Yorkshire and Northumberland. Coal Measures and Millstone Grit are an Upper Carboniferous sandstone and Carboniferous Limestone is a Lower Carboniferous sandstone. Both Upper and Lower Carboniferous sandstones have been used at Durham. Coal Measures can be found right in Durham County and was used for much of the restoration work. Sandstone from the Kepier Quarry, which is only a few miles away to the north-east, is a Coal Measures sandstone that was also chosen for restoration work at Durham Castle. Another Coal Measures sandstone used frequently for restoration was Dunhouse Stone from the Dunhouse quarry, near Darlington, that opened in 1926.

Dunhouse Stone is a fine to medium grained sandstone that was supplied in large blocks for restoration work. It is known to be of a creamy buff color and suitable for ashlar, carving, components, walling block and landscaping. It also is able to have a variety of finishes such
as dressed, fine rubbed, sawn and split. Dunhouse Quarry Company references Dunhouse Stone as a fine grained sandstone having excellent weathering properties, excellent resistance to pollution, a density of approximately 2400-2600 kg/m3, a porosity of 16%, and a compressive strength of 55.49 N/mm².

Of the Lower Carboniferous sandstones used, Black Pasture Stone is located in quarries near Chollerford, Northumberland. This sandstone was also used for restorations at Durham. Black Pasture Stone was used primarily before Dunhouse Stone and was considered a tough, fine-grained, buff colored sandstone.

Although various quarries have been used for construction and restoration work at Durham Castle, all of the sandstones selected have been locally quarried and readily available, which is still the case to date. The sandstones used also seem to have the same properties of color, cutting and carving abilities, as well as their weathering tendencies.

Using Folk’s categorization approach, the original sandstone used at Durham would be considered an immature stage of sedimentation, based upon its cementation matrix, and the sorting and roundness of its grains.

To understand the properties and composition of the sandstone, several
Table 5.01. Durham sandstone samples matrix.

<table>
<thead>
<tr>
<th>Stone</th>
<th>Description</th>
<th>Approx. Dimensions</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stone a</td>
<td>Weathered stone containing alveolar erosion</td>
<td>13&quot; x 5 1/2&quot; x 5&quot;</td>
</tr>
<tr>
<td>Stone b</td>
<td>Weathered stone containing alveolar erosion</td>
<td>10&quot; x 8&quot; x 7.5&quot;</td>
</tr>
<tr>
<td>Stone c</td>
<td>Replacement stone</td>
<td>10&quot; x 4 1/2&quot; x 2 1/2&quot;</td>
</tr>
</tbody>
</table>
methods and analysis techniques were conducted on both weathered and unweathered samples of the stone including optical microscopy, gravimetric analysis, soluble salt content, porosity and water absorption testing, scanning election microscopy and x-ray diffraction. [Table 5.01]

composition

gravimetric analysis

Gravimetric analysis by acid digestion was performed on Durham sandstone and on mortar samples at the start of the study to determine the approximate acid soluble component present in both the stone and the mortar used on Durham Castle. [Appendix F.01-F.06] The test was conducted on a sandstone sample; on the replacement pointing mortar seen on the east façade; and, on historic mortar found on the west façade. [Figure 5.01] [Appendix G.01-G.03]

This laboratory experiment is a simple method used to determine the three main components of a building stone or mortar: the fines, the acid soluble fraction, i.e., the calcareous binder; and, the acid insoluble fraction composed of siliceous materials. This experiment is a first approach to characterize the samples.

The sample is first ground into a powder, dried and weighed. A small
Figure 5.01 gravimetric analysis performed on Durham sandstone, a historic mortar and a current mortar.
amount of hydrochloric acid is then poured onto the sample to determine its reactivity with acid. The percentage of the sample that dissolves with hydrochloric acid corresponds to the acid soluble compounds present, such as calcium carbonate. Once this step is completed, the sample is poured into distilled water and stirred for 24 hours. Following the stirring, the sample is filtered through a filter paper to separate the clay fines in the sample from the aggregate or stone grains. The filter paper is then weighed to determine the clay fines present in the sample and the aggregate is sieved and weighed to determine the amount of siliceous minerals or aggregate and their particle size range.

Gravimetric analysis of the sandstone showed that the stone is composed mostly of sand particles, 64%; with a relative high percentage of clay fines, 32%; and, a small percentage of acid soluble minerals, 4%, most likely calcite, CaCO₃. The acid insoluble stone particles were mostly composed of smaller sand particles, most within the 100-200μm particle size range. [Appendix F.02]

The mortar found on the east façade is the same throughout the elevation and was introduced during the restoration campaign of the 1930s. The mortar has a hardness of 7 based on Mohs hardness scale and is composed of large size aggregate particles. The mortar is mostly intact on the façade and is only missing in areas of high activity, such as doorways. Gravimetric analysis determined the mortar to be composed
of 53% aggregate, 37% acid soluble fraction and 10% fines. The majority of aggregate size in the mortar ranges from 16-100μm. [Appendix F.04] The well-graded aggregate allows for low porosity.

The last sample used for gravimetric analysis was a mortar found on the terrace of the Castle. This sample was taken specifically to compare to the mortar used for repointing on the east façade. The historic mortar was comprised of 46% acid soluble fraction, 36% aggregate with an average particle range from 50-200μm, and 18% fines. [Appendix F.06] The high percentage of acid soluble material in the mortar suggests a lime binder. Table 5.02 summarizes the data obtained from all the three samples by gravimetric analysis.

Table 5.02. Summary of results from gravimetric analysis.

<table>
<thead>
<tr>
<th></th>
<th>Fines</th>
<th>Acid Soluble</th>
<th>Course</th>
</tr>
</thead>
<tbody>
<tr>
<td>Durham Sandstone -</td>
<td>32</td>
<td>4</td>
<td>64</td>
</tr>
<tr>
<td>Sample from Stone A</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Current Mortar - East</td>
<td>10</td>
<td>37</td>
<td>53</td>
</tr>
<tr>
<td>Façade Mortar</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Historic Mortar -</td>
<td>18</td>
<td>46</td>
<td>36</td>
</tr>
<tr>
<td>Terrace Mortar</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
soluble salt content

Efflorescence were only occasionally noted on the façade. However, research on alveolar erosion weathering shows that salts can be an important factor in their formation; therefore it was important to determine the concentration of salts that might be found in the sandstone. The test was conducted by following Dr. Charola’s determination of moisture and soluble salt content lab prepared for the University of Pennsylvania’s Advanced Conservation Science course. [Appendix G.04-G.07] Both a deteriorated stone sample (Stone B) and a replacement stone sample (Stone C) were used to determine soluble salt content.

The samples were first weighed, placed in an oven for 2 hours and removed, placed in a dessicator, and weighed again. This process was continued until the sample had two consecutive weighings less than 0.01% of the weight of the sample. The difference between this weighing and the initial weight of the sample then allowed to calculate the moisture content present in the stone. The weathered sandstone (Stone B) had a moisture content of 0.39% and the replacement stone (Stone C) had a moisture content of 0.14%. The samples were then ground into a uniform powder, weighed, and put in a beaker with distilled water. These suspensions were then stirred for 2 hours and left to settle overnight. Once the suspension had settled, it was filtered and the filter paper with solid residue was kept to calculate the weight of
soluble salt content and the solution obtained from filtering was kept to test for semi-quantitative analysis of soluble salts in the sample.

The total salt content in each sample was of 0.95% for sample B, the weathered sandstone, and 0.68% for sample C, the replacement stone. This suggests that the salt content is related to the length of exposure time.

To identify the salt content by type, the samples were tested using EM Quant ion salt strips, a semi-quantitative method for determining the specific ion content in the solution. [Figure 5.02] The samples were tested for chlorides, sulfates and nitrates by immersing the test strips in the sample solutions. Both samples came up positive for sulfates at a level between 200 and 400 mg/L. This concentration was then used to determine the concentration of sulfate present. The weathered stone had a sulfate ion percentage that ranged between 0.34% and 0.67% by weight, while that for the replacement stone ranged between 0.26% and 0.53% by weight. [Appendix F.07-F.08] Since the most likely salt to be accumulated would be gypsum, CaSO₄·2H₂O, due to air pollution, the estimated average gypsum content was calculated for both stones confirming that indeed gypsum accounts for all of the salt present in the stone. All the data are summarized in Table 5.03.
Figure 5.02. Testing for soluble salt content by volume using salt strips.
Table 5.03. Summary of the salt analysis for two samples of Durham Castle sandstone.

<table>
<thead>
<tr>
<th></th>
<th>% Moisture Content</th>
<th>% Soluble Salt Content</th>
<th>% Sulfate Ion Average</th>
<th>% Gypsum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Durham Sandstone -</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample from Stone B</td>
<td>0.39</td>
<td>0.95</td>
<td>0.34-0.67</td>
<td>0.91</td>
</tr>
<tr>
<td>Replacement Sandstone -</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample from Stone C</td>
<td>0.14</td>
<td>0.68</td>
<td>0.26-0.53</td>
<td>0.70</td>
</tr>
</tbody>
</table>

**porosity and water absorption and loss**

**water absorption/drying behavior**

Water absorption of the sandstone was tested using an unweathered sample and one with alveolar deterioration. This analysis was performed using Charola's Water Absorption and Drying Behavior Lab for Advanced Conservation Science. [Appendix G.08-G.10] This laboratory exercise incorporates a variety of standards including NORMAL 11/85, 7/81 and 29/88; ASTM C67-97 and C948-94; and the ICCROM ARC Laboratory Handbook. These are all incorporated to allow for the determination of capillary water absorption, water absorption by total immersion and porosity, and the drying rate in one exercise.

The analysis was carried out on a less weathered stone sample and a stone sample with alveolar erosion that were cut into 5 cm side cubic
samples. [Appendix F.09-F.12] The samples were placed in a container with distilled water only touching the bottom face. The samples were then weighed over a sequence of time as they absorbed water. [Chart 5.01] It took the unweathered sample 18 hours to reach an asymptotical water absorption value while the weathered sample required only 12 hours. The unweathered sample had an initial absorption rate of 0.00019g/g·sec0.5 while the deteriorated sample had an initial absorption rate of 0.00038g/g·sec0.5. The initial rate of absorption was much faster for the deteriorated sample, twice as fast, indicating that it had larger pores although the maximum of water absorbed was similar for both samples, about 22.2 grams.

chart 5.01. water absorption of durham sandstone samples.
Following the initial water absorption test, the sample was fully immersed in water for 24 hours to find out the maximum water absorption value. When this was determined, the sample was then taken out of the water and weighed over a sequence of time as it dried to determine the drying rates of the stone. The samples took a significantly longer amount of time to dry than they did to absorb water.

From the weight change of the samples, from dry weight to 24 hour water absorption, the apparent porosity percentage can be calculated. The unweathered sample absorbed 23.23 g of water while the weathered one absorbed 23.15 g. The difference between the maximum amount of water absorbed by capillarity and that by total immersion is about 1 g, for either sample. This indicates that there are some large pores that require total immersion to be completely filled and that the overall porosity of the stone has not been affected by the weathering, rather, the changes that are induced by the weathering are concentrated on the surface.

The Apparent Porosity for these samples was calculated to be 5.71% and 5.88% for the unweathered and the weathered sample, respectively. The Open Porosity of the samples is calculated based on the ratio of open pores in the sample to its total volume. For the unweathered sample, the Open Porosity was 18.58 % while it was 18.52 % for the weathered sample. This value is probably smaller than the actual value since the
calculated volume of the weathered sample was approximated to that of a perfect cube, while in reality it is likely smaller. The summary of the complete water absorption data is shown in Table 5.04.

Table 5.04. Summary of Water Absorption.

<table>
<thead>
<tr>
<th>Water Absorption</th>
<th>Capillarity</th>
<th>24 hour immersion</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Initial Absorption Rate (g/g•sec^{0.5})</td>
<td>Maximum Water Absorbed</td>
</tr>
<tr>
<td>Unweathered Sample</td>
<td>0.00019</td>
<td>22.16</td>
</tr>
<tr>
<td>Alveolar Erosion Sample</td>
<td>0.00038</td>
<td>22.19</td>
</tr>
</tbody>
</table>

The drying curves are shown in Chart 5.02. The initial drying rate is similar for both weathered and unweathered stones. However, it can be seen that the weathered stone takes longer to dry than the unweathered one. This reflects the relative increased clay content in the weathered stone as the calcitic binder is lost and gypsum is formed. It is known that gypsum tends to block pores retaining moisture, therefore, the weathering process is exacerbated in weathered samples—moisture enhancing clay expansion—as they take longer to dry.
chart 5.02. Drying curve of Durham sandstone samples.
optical microscopy

Thin sections were prepared from two samples: a weathered stone (Stone A) and a replacement stone (Stone C) were selected for sampling. Of the weathered stone, two separate samples were taken from a less weathered area and from a cavity while only one sample was taken from the replacement stone. The samples were cut in cross-section so that when mounted the sample would contain the inner stone as well as the exterior face to determine any differences in the structure. When the samples were sectioned and mounted they were injected with blue epoxy to make any porosity or variances in the structure clearer. While the stones are all sandstone quarried from the same place, differences can be noted when looking at the thin sections.

Stone A had specimens taken both from a less weathered area (A_A) and from a cavity (A_C). Looking at the less weathered area it can be seen that the stone is composed of quartz grains (43%), silica, clay matrix (14%) and in some areas, small amounts of mica. [Figure 5.03] The apparent porosity in the sample is 13%. [Figures 5.04-5.07] As the sample is examined from the inner portion of the stone towards the exterior face, there is a higher level of porosity, indicating the weathering occurring from the surface of the stone. [Figure 5.08-5.09] The quartz grains in the sandstone are poorly graded, of angular and sub-angular shape with an average grain size of 100μm. Throughout the sample there is a high amount of inter-granular cracking that occurs between
figure 5.03. stone sample A thin section. 1.0x magnification.
figure 5.04. stone sample A.

figure 5.05. stone sample A porosity, 13%

figure 5.06. stone sample A quartz, 43%

figure 5.07. stone sample A clay, 14%
figure 5.08. stone sample A surface. 3.0x magnification.

figure 5.09. stone sample A interior. 3.0x magnification.
the quartz grains in the stone. The clay matrix can be seen surrounding the quartz grains as well as silica. After disintegration of the grains by weathering and compaction, silica forms around the grains. However, in this sandstone, the silica content is not enough to bond the grains together.

When looking at the cavity sample (A_C) of the weathered stone A, significant changes can be seen. [Figure 5.10] The cavity of the stone has a higher level of porosity, about 28%. It also contains a significant amount of micro-cracking throughout the entire sample. The cavity contains a higher percent of clay matrix than that of the un-weathered stone as well, approximating 19%. [Figures 5.11-5.14] The cavity of Sample A also has extreme differences between the surface of the stone and the interior. The surface of the cavity being very friable, indicating disintegration of the surface. [Figure 5.15-5.16]

The replacement stone currently used at Durham Castle is surprisingly more porous than the original stone samples. [Figure 5.17] Mineralogically it is similar to the current stone, containing a noticeable amount of clay (20%), silica and quartz grains (60%). The sample is comprised of sub-rounded and sub-angular grains averaging 100μm in size with an average porosity of 20% throughout the sample. [Figures 5.18-5.21] The replacement stone does not have a lot of differences between the surface and interior because it has not weathered as the other samples. [Figures 5.22-5.23]
Figure 5.10. Stone sample A cavity thin section. Note increased porosity (> blue) at top (surface). 1.0x magnification.
Figure 5.11. Stone sample A cavity

Figure 5.12. Stone sample A cavity porosity, 28%

Figure 5.13. Stone sample A cavity quartz, 40%

Figure 5.14. Stone sample A cavity clay, 19%
Figure 5.15 shows stone sample A cavity surface at 3.0x magnification.

Figure 5.16 shows stone sample A cavity interior at 3.0x magnification.
Figure 5.17. Stone sample C thin section - replacement stone. 1.0x magnification.
figure 5.18. stone sample C
figure 5.19. stone sample C porosity, 20%
figure 5.20. stone sample C quartz, 60%
figure 5.21. stone sample C clay, 20%
figure 5.22 stone sample C surface - replacement stone surface. 3.0x magnification.

figure 5.23 stone sample C surface - replacement stone interior. 3.0x magnification.
Table 5.05 summarizes this data from optical microscopy, listing the differences between the samples.

Table 5.05. Summary of data obtained by image analysis with optical microscopy

<table>
<thead>
<tr>
<th></th>
<th>Porosity %</th>
<th>Quartz %</th>
<th>Clay Matrix %</th>
<th>Average Grain Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample A</td>
<td>19</td>
<td>61</td>
<td>20</td>
<td>100μm</td>
</tr>
<tr>
<td>Sample A_C</td>
<td>36</td>
<td>40</td>
<td>24</td>
<td>100μm</td>
</tr>
<tr>
<td>Sample C</td>
<td>20</td>
<td>60</td>
<td>20</td>
<td>100μm</td>
</tr>
</tbody>
</table>

The effects of deterioration are noticeable in the thin sections. The original stone has a high porosity and clay matrix but weathered zones (A_C) display a twofold increase in porosity. Based on the high percent of clays present, determining the type of clay through x-ray diffraction is fundamental in determining how it may be affecting stone performance.
After microscopic examination was performed on the samples, Scanning Electron Microscopy and X-Ray Diffraction were performed to determine the elemental and mineralogical composition of the samples, respectively. Scanning Electron Microscopy was performed on three samples from Stone A.

Sample A1 was taken from a portion of stone that had no sign of weathering. This served as a reference for comparison with the weathered samples. The sand grains appear to be only slightly separated and pores are evident throughout the sample. [Figure 5.24] Some larger calcite crystals are also present. The most striking feature is the abundance of clay tablets and other flaky minerals composed of mica.\textsuperscript{13} While the grains are apparent in the stone, it is the clays that are dominant in the sample. [Figures 5.25-5.27]

Sample A2 was taken from a portion of the stone containing alveolar erosion. The sample was taken strategically so that the edges of the sample are segments along the formed cavities, and the center of the sample is a hardened space between two cavities. [Figure 5.28]
figure 5.24. unweathered sample, A1, showing the grain structure and porosity of the sandstone.
figure 5.25. unweathered sample, A1, showing the quartz grains, calcite and porosity of the sandstone.
Figure 5.26. Unweathered sample, A1, showing the flakey clays dominant in the stone.
figure 5.27. unweathered sample, A1, showing the formed clays in the stone.
Figure 5.28. Alveolar erosion sample, A2, for scanning electron microscopy.
Looking at the differences in the sample between the edges and the center it can be seen that the center of the sample is much denser than the edges. The grains appear closer together and less pores are visible. [Figures 5.29] At high magnification, the clay stacks can be seen in both the edge of the sample and in the center. In the center of the stone, the clays are more organized than at the edges of the sample that correspond to the top of the lower alveole and to the bottom or the upper alveole. [Figures 5.30-5.31]

SEM of both samples was very informative, not only by visualizing the clay matrix but also by seeing the variation in the way the clay was stacked. In specific areas throughout the stone, the clays can be seen stacked into columns, i.e., as kaolinite does. By looking at the photos from SEM analysis it is apparent that clays are present throughout the entire stone but that they are weathering. These clays appear “flaky” and are not correctly stacked in columns. The edges of the clays also appear fragmented and appear white in SEM analysis which may be a result of edge charges.\textsuperscript{14} Gravimetric analysis and optical microscopy only determined 15-30\% of clay matrix in the stone; however, after performing SEM analysis it was apparent that clays were a much more dominant component in the sandstone than was initially seen. This can be attributed to the lower density that clays have with respect to quartz.
Figure 5.29. Center of alveolar sample, A2, showing more compaction.
Figure 5.30. Center of alveolar sample, A2, showing properly formed clays.
Figure 5.31. Edge of alveolar sample, A2, showing flaky clays and expanding clays.
The third, larger sample was used to do both SEM and elemental analysis by energy dispersive spectroscopy (SEM-EDS) to determine elemental differences in various areas of the stone. [Appendix F.13] First, two areas were chosen towards the center of the stone sample. Area 1 was a planar surface and area 2 was a flakier portion of the stone. [Figure 5.32] Area 1 was high in silicon and also contained sodium, magnesium, aluminum and calcium. [Chart 5.03] Area 2 had still a high amount of silicon, an increased amount of aluminum, and still contained sodium, magnesium and calcium. Unlike area 1, area 2 also had potassium present. [Chart 5.04]

After looking at two areas in the center of the stone, an area was chosen on the edge of the sample where alveolar erosion occurred. [Figure 5.33] The edge of the stone contained a high amount of silicon, lower carbon and calcium, indicating a decrease in the binding material, and contained aluminum and sodium. [Chart 5.05]

Noting the element change in different areas of the stone, elemental mapping was conducted to show the change in elements in specific areas across the sample. From the previous elemental analysis on specific portions, it was determined to perform elemental mapping for silicon, aluminum, calcium, carbon, potassium, sodium and magnesium elements. [Figures 5.34-5.35] The elemental mapping reflected the two
Figure 5.32. Center areas tested for SEM-EDS analysis.
chart 5.03. area one (flat surface) tested for sem-eds analysis.

chart 5.04. area two (flaky surface) tested for sem-eds analysis.
Figure 5.33. Edge area tested for SEM-EDS analysis.
chart 5.05. area of edge of stone where alveole occurs sem-eds analysis.
Figure 5.34. Elemental mapping of sample stone A
Figure 5.35. Elemental mapping of sample stone A cavity
areas that were previously studied. The elements silicon, aluminum, potassium, sodium and magnesium are all shown stronger in the elemental mapping of the cavity portion of the sample as expected. This shows the increase of these elements due to higher clay content and deterioration effects. Calcium and carbon, representing the calcium carbonate binding material of the stone, is reduced in the elemental mapping of the cavity, expressing disintegration.

**x-ray diffraction**

Following scanning electron microscopy, X-ray powder diffraction was used as an analytical method to determine the mineralogical composition of a powdered sample of stone. Since clays were largely seen in SEM analysis, it was important to identify the clay types present in the sandstone to begin to understand the effects they have on the nature of the stone. The sample was prepared by grinding a portion of stone with a mortar and pestle and stirring it on a stir plate for 2 hours with deionized water. Then it was left to settle overnight and filtered through an ashless filter paper to collect the solid. This powdered sample was then taken for x-ray diffraction analysis. The sample was placed as a powder in an aluminum mount.¹⁵

The spectrum was run and the results obtained by computer matching were analyzed to determine the minerals present in the sandstone.
[Appendix F.14-F.15] As expected, quartz was the dominant minerals in the sample as was clay. [Chart 5.06] Multiple clay minerals were found throughout the sample including illite, montmorillonite, dickite, albite, kaolinite, palygorskite and kyanite, in decreasing order of concentration. [Chart 5.07]

Mixed-layer clay minerals are interstratified layers of multiple kinds of clay minerals. X-ray diffraction detected an illite and montmorillonite mineral layer which contributes to approximately 20% of the stone sample’s composition. Illite is the most abundant clay mineral present in sedimentary rocks,\textsuperscript{16} which we find holding the highest percentage in this sandstone as well. It is a non-expanding phyllosilicate composed of tetrahedron – octahedron – tetrahedron layers with the interlayer space consisting of poorly hydrated potassium cations which usually prevent the clay from swelling. However, in this sample, illite is combined with montmorillonite which is another phyllosilicate. Montmorillonite is also a 2:1 layer structure which unlike illite, is highly expansive. These clays can form mixed layers of illite-montmorillonite, and they act as a swelling clay in the sandstone. For this reason, the swelling contraction cycles undergone by any clay, are significantly enhanced and explain in part the susceptibility of the sandstone to deterioration in the presence of moisture.
chart 5.06. xrd data showing the high quartz count in the sandstone.
chart 5.07. xrd data showing the amount of clays distributed throughout the sandstone.
Kaolinite is one of the most common clay mineral groups which belongs to the family of phyllosilicates (layer silicates) and is a 1:1 layer type mineral group.\textsuperscript{17} Kaolinites general composition is Al\textsubscript{2}Si\textsubscript{2}O\textsubscript{5}(OH)\textsubscript{4}. Kaolinite is always a secondary mineral that is formed by weathering or the hydrothermal alteration of aluminum silicates.\textsuperscript{18} Kaolinite is not a particularly expansive clay but also comprises a much lower count in the sandstone than illite-montmorillonite.

\textit{Analysis conclusions}

Table 5.06 compares the results of the different porosities measured by the different techniques.

<table>
<thead>
<tr>
<th>Porosity Comparisons</th>
<th>Apparent Porosity</th>
<th>Open Porosity %\textsubscript{v/v}</th>
<th>Porosity %\textsubscript{v/v}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity % reported by Dunhouse Stone</td>
<td>% w/w 24 hr Water Immersion</td>
<td>24 hr Water Immersion</td>
<td>Optical Microscopy</td>
</tr>
<tr>
<td>Unweathered Sample</td>
<td>6</td>
<td>19</td>
<td>19</td>
</tr>
<tr>
<td>Alveolar Erosion Sample</td>
<td>6</td>
<td>19</td>
<td>36</td>
</tr>
<tr>
<td>Replacement Stone</td>
<td>16</td>
<td></td>
<td>20</td>
</tr>
</tbody>
</table>

It can be seen that for the unweathered sample, the Open Porosity measured by the 24 hr total immersion corresponds to that assessed by optical microscopy. In the case for the weathered sample, the value was underestimated, as indicated previously, because of the approximation
of the sample volume used in the calculation. It becomes evident, that the porosity increases with weathering. The average porosity of the quarry stone is 16%, this increases to 19% or 20% for stone exposed but not yet evidently weathered, and this value is nearly doubled (36%) for the very weathered stone. It is clear that indication of whether the porosity is measured by volume or by weight, is critical.

Based upon the analysis and laboratory experiments done on the samples of Durham sandstone, it can be concluded that the stone deterioration pattern observed, the alveolar erosion, can be attributed to a large degree to the geo-chemical nature of the stone, and in particular the presence of expansive clays. Even unweathered portions of stone and replacement stone have high porosity and the clay matrix contributes to the weathering by its behavior in the presence of moisture. The likely presence of gypsum serves as a multiplying factor in the deterioration.

The grain sizes in the stone also play an important role in its deterioration. The particle size distribution of a stone can affect its permeability, porosity and water vapor transmission. Since the stone is composed mostly of fine sand particles and silt, it explains some of the deterioration mechanisms that are occurring in the stone. Fine sand and silt are useful in filling in voids between larger sized aggregate, however, if there are only smaller particles, they become
easily dislodged and transportable when in contact with water. This fact, in conjunction with the presence of expanding clays and gypsum, makes the sandstone extremely vulnerable to the climatic conditions which occur in Durham.

2 Ibid. p. 62
3 Ibid. p. 61
5 Folk, R.L. Stages of textural maturity in sedimentary rocks. p. 127.
7 Ibid. p. 70
8 Ibid. p. 70
10 Ibid.
12 Ibid. p. 66
13 These observations are based upon a discussion of the SEM results with John Walsh which occurred on April 8, 2010.
14 Ibid.
15 General Note: Preparation for x-ray diffraction containing clay minerals is a difficult process and there are several more in-depth ways to prepare samples which were not conducted here. To provide a better result of the clay matrixes that are found in the Durham sandstone, analysis should be done using preparation techniques discussed by Moore and Reynolds in X-Ray Diffraction and the Identification and Analysis of Clay Minerals, Chapter 5: Sample Preparation Techniques for Clay Minerals, 1989.
17 Ibid. p. 102.
Chapter 6: Conclusions/Recommendations

Conclusions

The study has focused on the sandstone masonry at Durham Castle including its history, repairs and restorations, and its current condition. In particular, it has focused on the deterioration phenomena known as alveolar erosion and its effect on the surface of Durham sandstone. From the documentation and material analysis conducted on the sandstone it can be determined that the stone’s deterioration tendencies are primarily a result of the geo-chemical nature of the sandstone. The weathering is then enabled by repeated contact with moisture and wind and accelerated by the presence of salts.

Researching case studies on alveolar erosion and performing analysis on the sandstone at Durham lead to the hypothesis that the prominent forming of alveolar erosion is a result of the high clay content of the sandstone and the presence of salts, with the formation of silica and the re-deposition of the calcitic binder acting as contributing factors.

Recommendations

Currently the only form of repair at Durham Castle is the total replacement of stone in kind. To maximize the maintenance of original stone on the structure, preventative treatments are advised to reduce the amount
of replacement. Therefore, to arrest the formation of alveolar erosion in the sandstone, several recommendations are suggested including the removal or immobilization of salts, a treatment to aid in the anti-swelling of clays and finally, a consolidant treatment.

1. Salt extraction versus Barium hydroxide to block sulfates in stone
Salt removal treatments should be studied and implemented to extract the sulfates from the sandstone. Extraction of the sulfates should be done prior to any strengthening treatments done on the stone. However, since the salts are present throughout the stone an alternative solution would be the use of barium hydroxide to block the movement of sulfates by the formation of the extremely insoluble barium sulfate. Barium hydroxide can be applied in various ways and this needs to be explored on site.

2. Anti-swelling agent
One of the most significant problems with the Durham sandstone is its high clay content. It is advised that an anti-swelling agent be applied prior to a consolidation treatment as a remedial method to reduce the deterioration problem. An anti-swelling agent is a preservative that stops the hygroscopic swelling of clays located in a stone. Swelling and shrinking that occurs in the clays of the sandstone are a result of wetting and drying cycles occurring on the stone. The agent’s action is based on
replacing the interlayer ions that are the ones originating the swelling. Antihygro – an anti-swelling agent manufactured by Remmers, is a well known treatment that binds clay and decreases swelling by up to 50% without altering the materials absorption and breathing properties.¹

3. Consolidant Treatment
The function of a consolidant treatment at Durham would be to reestablish the inter-granular cohesion of the sandstone that has become friable and deteriorated by granular disintegration. The major principles when deciding on a consolidant treatment are significant penetration, adhesion, gelation and drying properties, have a low modulus of elasticity and be carried in organic solvents which will not cause the swelling of clays.²

Ethyl silicates are often used in materials with high clay content for they deposit silica gel as a binder into deteriorated stonework. The silica binder gel replaces the original binder that has been lost through weathering. Elastified Silicate-Esters are silicate-esters that introduce an increase in E-modulus allowing for expansion to occur. Since an anti-swelling agent would be implemented prior to a consolidant, elastified silicate-esters may not be necessary but they still may be the better option considering the friability and nature of the stone. Recommended consolidants for evaluation include Remmers SAE 300E and Prosoco OH100.
These recommendations should be investigated further to determine the best method for application to the sandstone at Durham. They should be executed in a laboratory setting so that equivalent tests as those performed throughout this study can be carried out again on the treated stone to measure the efficacy of the treatments. Following laboratory analysis and conclusions, these treatments should be applied on site at Durham and monitored before any full implementation of the treatments is considered.

1 Remmers, Funcosil Antihygro Technical Information Sheet.


“Engineered Weather Data for Flyingdales, UK.”


Prosoco, Inc. 2010. Material Safety Data Sheet for Consevare OH100.
Remmers, Ltd. 2006. Material Safety Data Sheet for Funcosil Antihygro.

Remmers, Ltd. 2006. Material Safety Data Sheet for SAE 300E.


Durham University Library, Archives and Special Collections. “Durham Castle buildings archive.”


appendix a. methodology.
research

- castle history
- context
- alveolar erosion

documentation

- pilot facade survey

material analysis

- petrographic analysis
- laboratory testing
- scanning electron microscopy
- x-ray diffraction

conclusions and recommendations
appendix b. castle drawings.
Appendix C. Conditions Glossary.
CONDITIONS GLOSSARY

TYPICAL EXAMPLE

TOTAL REPAIR

THE COMPLETE UNIT REPLACEMENT OF STONE

SYMBOLOGY

TYPICAL EXAMPLE
CONDITIONS GLOSSARY

PARTIAL REPAIR

PARTIAL REPLACEMENT OF STONE IN KIND

SYMBOLS

TYPICAL EXAMPLE
PATCHING

STONE SURFACE REPLACEMENT WITH MORTAR

SYMBOLOGY

TYPICAL EXAMPLE
METALLIC ELEMENTS

ALL METALLIC INSERTIONS EMBEDDED INTO THE MASONRY WALL

SYMBOLOGY

TYPICAL EXAMPLE
ALVEOLIZATION

FORMATION ON THE STONE SURFACE OF CAVITIES OF VARIABLE SHAPES AND SIZES

SYMBOLOGY

MILD

MODERATE

SEVERE

C.05
MINOR CRACKING

FRACTURES VARYING IN ORIENTATION, DEPTH, AND LESS THAN 1/8” IN WIDTH
MAJOR CRACKING

FRACTURES VARYING IN ORIENTATION, DEPTH, AND GREATER THAN 1/8” IN WIDTH

SYMBOLOGY

TYPICAL EXAMPLE
CONDITIONS GLOSSARY

PEELING [DETACHED]

THIN DELAMINATION OF TOOLED AND PATCHED STONE SURFACE

SYMBOLOGY

MILD

MODERATE

SEVERE

C.08
CONDITIONS GLOSSARY

PEELING [GONE]

THIN DELAMINATION OF TOOLED AND PATCHED STONE SURFACE RESULTING IN LOST STONE

SYMBOLOGY

MILD

MODERATE

SEVERE

C.09
FRIABILITY

DISAGGREGATION OF STONE SURFACE USUALLY IN ASSOCIATION WITH MODERATE AND MAJOR LOSS

SYMBOLOGY

MILD

MODERATE

SEVERE

C.10
CONDITIONS GLOSSARY

DETACHMENT [VISIBLE]

PHYSICAL SEPARATION OF STONE INTO LAYERS

SYMBOLOGY

MILD

MODERATE

SEVERE

C.11
CONDITIONS GLOSSARY

DETACHMENT [BLIND]

PHYSICAL SEPARATION OF STONE OCCURING BENEATH THE STONE AND NOT Visible

SYMBOLS

TYPICAL EXAMPLE
LINEAR EROSION

STRIATED LOSS OF STONE SURFACE RELATED TO SEDIMENTARY BEDDING

SYMBOLOGY

- MILD
- MODERATE
- SEVERE

C.13
CONDITIONS GLOSSARY

LOSS [MODERATE]

< 1/2” OF MISSING STONE

SYMBOLOGY

TYPICAL EXAMPLE
CONDITIONS GLOSSARY

LOSS [MAJOR]

> 1/2" OF MISSING STONE

SYMBOLOGY

TYPICAL EXAMPLE
CONDITIONS GLOSSARY

CONTOUR SCALING

DETACHMENT OF STONE IN STACKED LAYERS REMEMBERING TOPOGRAPHY, OFTEN IN ASSOCIATION WITH VISIBLE AND BLIND DETACHMENT

SYMBOLOGY

MILD

MODERATE

SEVERE
CONDITIONS GLOSSARY

FLAKING

DETACHMENT OF SMALL, THIN FLAT OR CURVED SCALES ON THE STONE SURFACE

SYMBOLOGY

MILD

MODERATE

SEVERE

C.17
CONDITIONS GLOSSARY

DISPLACEMENT

MOVEMENT OF STONE FROM ORIGINAL POSITION

SYMBOLOGY

+  -

TYPICAL EXAMPLE
EFFLORESCENCE

WHITISH, POWDERY CRYSTALS ON THE STONE SURFACE MADE OF SOLUBLE SALT

SYMBOLOGY

MILD

MODERATE

SEVERE

C.19
SPALLING

IRREGULAR LOSS USUALLY FROM IMPACT OR CORROSION

SYMBOLOGY

MILD

MODERATE

SEVERE

C.20
MISSING JOINTS

MISSING OR DAMAGED JOINTS

SYMBOLOGY

MILD

MODERATE

SEVERE

C.21
CONDITIONS GLOSSARY

INTERFACE EROSION

EROSION OCCURRING AT THE INTERFACE OF ORIGINAL STONE AND STONE REPAIR

SYMOLOGY

TYPICAL EXAMPLE
CONDITIONS GLOSSARY

METALLIC STAINING

RED-BURN DISCOLORATION RELATED TO IRON CORROSION

SYMBOLOGY

MILD

MODERATE

SEVERE

C.23
CONDITIONS GLOSSARY

VEGETATION

THE PRESENCE OF HIGHER PLANT FORMS, INCLUDING THEIR ROOT REMNANTS

SYMBOLOGY

TYPICAL EXAMPLE
GUANO

BIRD EXCREMENT

SYMBOLOGY

MILD

MODERATE

SEVERE

C.25
CONDITIONS GLOSSARY

MICRO-FLORA

THE PRESENCE OF ALGAE AND FUNGI IDENTIFIED AS LOCALIZED AREAS OF BLACK, GREEN, OR BROWN DISCOLORATION

SYMBOLOGY

C.26
appendix d. condition survey.
appendix e. deterioration mechanisms/assessments.
Treatment and flaking
ty and detachment
appendix f. analysis data.
## ARCHITECTURAL CONSERVATION LABORATORY
### UNIVERSITY OF PENNSYLVANIA

### STONE ANALYSIS BY ACID DIGESTION

**Project/Site:** Durham Castle  
**Location:** Durham, England  
**Date Sampled:** July 28, 2009  
**Analysis Performed By:** Tiffani Simple  
**Date Analyzed:** November 17, 2009

### DESCRIPTION OF SAMPLE

**Type/Location:** Sandstone  
**SAMPLE No.** Stone A  
**Surface Appearance:** friable  
**Cross Section:**  
**Color:** 2.5Y 7/3 Pale Yellow  
**Texture:**  
**Hardness:** 4  
**Gross Wgt.:** 26.82

### COMPONENTS

<table>
<thead>
<tr>
<th>Fines</th>
<th>Color: 2.5Y8/1 White</th>
<th>Wgt.: 8.57g</th>
<th>Wgt. %: 32</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Matter:</td>
<td>Clays</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acid Soluble Fraction:</td>
<td>Wgt: 0.99g</td>
<td>Wgt. %: 4</td>
<td></td>
</tr>
<tr>
<td>Desc. Of reaction:</td>
<td>reactive</td>
<td>Filtrate Color:</td>
<td>yellow</td>
</tr>
<tr>
<td>Composition:</td>
<td>Calcareous binder</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Acid Insoluble Fraction:</td>
<td>Color: 2.5Y 7/2 Light Gray</td>
<td>Wgt.: 17.26</td>
<td>Wgt. %: 64</td>
</tr>
<tr>
<td>Grain Shape:</td>
<td>angular and sub-angular</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mineralogy:</td>
<td>mostly silica sand, quartz</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Sieve analysis:

<table>
<thead>
<tr>
<th>Screen</th>
<th>% Retained</th>
</tr>
</thead>
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<tr>
<td>8</td>
<td>1.53</td>
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<tr>
<td>16</td>
<td>0.29</td>
</tr>
<tr>
<td>30</td>
<td>0</td>
</tr>
<tr>
<td>50</td>
<td>1.00</td>
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<tr>
<td>100</td>
<td>64.48</td>
</tr>
<tr>
<td>200</td>
<td>22.77</td>
</tr>
<tr>
<td>pan</td>
<td>9.91</td>
</tr>
</tbody>
</table>

### ASSESSMENT

**Type:**  
Fines: mostly clays  32%  
Acid Soluble: calcareous material, possibly CaCO3  4%  
Stone particles: mostly silica sand and quartz:  64%  
Total  99.99%
**ARCHITECTURAL CONSERVATION LABORATORY**
**UNIVERSITY OF PENNSYLVANIA**

**MORTAR FROM EAST FAÇADE, 1930S RESTORATION**

<table>
<thead>
<tr>
<th>Project/Site: Durham Castle</th>
<th>Date Sampled: July 28, 2009</th>
</tr>
</thead>
<tbody>
<tr>
<td>Location: Durham, England</td>
<td>Date Analyzed: November 17, 2009</td>
</tr>
<tr>
<td>Analysis Performed By: Tiffani Simple</td>
<td></td>
</tr>
</tbody>
</table>

**DESCRIPTION OF SAMPLE**

<table>
<thead>
<tr>
<th>Type/Location: Mortar/Courtyard</th>
<th>SAMPLE No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Appearance: large aggregates, non-cohesive</td>
<td></td>
</tr>
<tr>
<td>Cross Section:</td>
<td></td>
</tr>
<tr>
<td>Color: 5YR 6/2 Pinkish Gray</td>
<td>Texture:</td>
</tr>
<tr>
<td>Hardness: 7</td>
<td>Gross Wgt.: 16.60</td>
</tr>
</tbody>
</table>

**COMPONENTS**

<table>
<thead>
<tr>
<th></th>
<th>Color: 5YR 7/1 Light Gray</th>
<th>Wgt.: 1.66</th>
<th>Wgt. %: 10</th>
</tr>
</thead>
<tbody>
<tr>
<td>Organic Matter:</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Composition:</td>
<td>clays</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Wgt.: 6.13</th>
<th>Wgt. %: 37</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acid Soluble Fraction:</td>
<td>Desc. Of reaction: highly reactive</td>
<td>Filtrate Color: yellow</td>
</tr>
<tr>
<td>Composition:</td>
<td>CaCO₃</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>Color: 5YR 6/2 Reddish Gray</th>
<th>Wgt.: 8.79</th>
<th>Wgt. %: 53</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain Shape:</td>
<td>sub-rounded and rounded</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mineralogy:</td>
<td>quartz, sand particles</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sieve analysis:</th>
<th>Screen</th>
<th>% Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>0.68</td>
<td></td>
</tr>
<tr>
<td>16</td>
<td>31.97</td>
<td></td>
</tr>
<tr>
<td>30</td>
<td>27.42</td>
<td></td>
</tr>
<tr>
<td>50</td>
<td>20.48</td>
<td></td>
</tr>
<tr>
<td>100</td>
<td>14.11</td>
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</tr>
<tr>
<td>200</td>
<td>4.21</td>
<td></td>
</tr>
<tr>
<td>pan</td>
<td>1.14</td>
<td></td>
</tr>
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</table>

**ASSESSMENT**

<table>
<thead>
<tr>
<th>Mortar Type:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fines: clays, silica sand 10%</td>
</tr>
<tr>
<td>Acid Soluble: calcareous binder, 37%</td>
</tr>
<tr>
<td>Aggregate: mostly quartz, 53%</td>
</tr>
</tbody>
</table>
Mortar from East Facade, 1930s
Gravimetric Analysis

Mortar from East Facade, 1930s
Particle Size Distribution
**ARCHITECTURAL CONSERVATION LABORATORY**
**UNIVERSITY OF PENNSYLVANIA**

**HISTORIC MORTAR FROM TERRACE ANALYSIS**

<table>
<thead>
<tr>
<th>Project/Site: Durham Castle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Location: Durham, England</td>
</tr>
<tr>
<td>Date Sampled: July 28, 2009</td>
</tr>
<tr>
<td>Analysis Performed By: Tiffani Simple</td>
</tr>
<tr>
<td>Date Analyzed: November 17, 2009</td>
</tr>
</tbody>
</table>

**DESCRIPTION OF SAMPLE**

<table>
<thead>
<tr>
<th>Type/Location: Mortar/Terrace</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAMPLE No.</td>
</tr>
<tr>
<td>Surface Appearance:</td>
</tr>
<tr>
<td>Cross Section:</td>
</tr>
<tr>
<td>Color: 2.5Y 5/1 Gray</td>
</tr>
<tr>
<td>Hardness: 2</td>
</tr>
<tr>
<td>Gross Wgt.: 17.06</td>
</tr>
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</table>

**COMPONENTS**

<table>
<thead>
<tr>
<th>Components</th>
<th>Color: 2.5Y 6/2 Light Brownish Gray</th>
<th>Wgt.: 3.09</th>
<th>Wgt. %: 18</th>
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</thead>
<tbody>
<tr>
<td>Fines:</td>
<td>Organic Matter: Hair</td>
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<tr>
<td>Acid Soluble Fraction:</td>
<td>Wgt: 7.79</td>
<td>Wgt. %: 46</td>
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</tr>
<tr>
<td>Aggregate:</td>
<td>Desc. Of reaction: highly reactive</td>
<td>Filtrate Color: yellow</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Composition: calcareous binder</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Color: 2.5Y 7/1 Light Gray</td>
<td>Wgt.: 6.16</td>
<td>Wgt. %: 36</td>
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<tr>
<td></td>
<td>Grain Shape: sub-angular and sub-rounded</td>
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<td></td>
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<tr>
<td></td>
<td>Mineralogy: silica sand</td>
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**Sieve analysis:**

<table>
<thead>
<tr>
<th>Screen</th>
<th>% Retained</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
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</tr>
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<td>16</td>
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</tr>
<tr>
<td>30</td>
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<td>50</td>
<td>18.83</td>
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<tr>
<td>100</td>
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<tr>
<td>200</td>
<td>22.08</td>
</tr>
<tr>
<td>pan</td>
<td>9.90</td>
</tr>
</tbody>
</table>

**ASSESSMENT**

Mortar Type:

- Fines: mostly clay, 18%
- Acid Soluble: calcareous binder, 46%
- Aggregate: mostly silica sand particles, 36%
Historic Mortar from Terrace
Gravimetric Analysis

Particle Size Distribution

Historic Mortar from Terrace
Particle Size Distribution
### Soluble Salt Data

#### Sample B (Current Stone)

<table>
<thead>
<tr>
<th></th>
<th>Sample C (Replacement)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wh</td>
<td>38.07</td>
</tr>
<tr>
<td>Wh + Sample</td>
<td>53.35</td>
</tr>
<tr>
<td>We - Sample</td>
<td>15.28</td>
</tr>
<tr>
<td>Wh + Wd (Sample Dry)</td>
<td>53.29</td>
</tr>
<tr>
<td>Wd - Sample Dry</td>
<td>15.22</td>
</tr>
</tbody>
</table>

#### % Moisture Content (w/w) = \( \frac{(we - wd)}{wd} \times 100 \)

<table>
<thead>
<tr>
<th></th>
<th>Sample C (Replacement)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wh</td>
<td>38.21</td>
</tr>
<tr>
<td>Wh + Sample</td>
<td>53.01</td>
</tr>
<tr>
<td>W'd (Sample Dry)</td>
<td>14.80</td>
</tr>
<tr>
<td>Wp</td>
<td>4.17</td>
</tr>
<tr>
<td>Wg</td>
<td>94.01</td>
</tr>
<tr>
<td>Wg + wetf</td>
<td>129.41</td>
</tr>
<tr>
<td>Wg + dryf</td>
<td>112.84</td>
</tr>
<tr>
<td>Wdryf</td>
<td>18.83</td>
</tr>
</tbody>
</table>

#### % Soluble Salt Content (w/w) = \( \frac{(w'd - wdryf - wp)}{w'd} \times 100 \)

<table>
<thead>
<tr>
<th></th>
<th>Sample C (Replacement)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloride Test (Cl(^{-}))</td>
<td>0 mg/L</td>
</tr>
<tr>
<td>Sulfate Test (SO(_4^{2-}))</td>
<td>200 &gt; 400 mg/L</td>
</tr>
<tr>
<td>Nitrate Test (NO(_2^{-}))</td>
<td>0 mg/L</td>
</tr>
<tr>
<td>Nitrate Test (NO(_3^{-}))</td>
<td>0 mg/L</td>
</tr>
<tr>
<td></td>
<td>% Moisture Content</td>
</tr>
<tr>
<td>------------------</td>
<td>--------------------</td>
</tr>
<tr>
<td>Durham Sandstone - Sample from Stone B</td>
<td>0.39</td>
</tr>
<tr>
<td>Replacement Sandstone - Sample from Stone C</td>
<td>0.14</td>
</tr>
<tr>
<td>Gypsum is CaSO$_4 \cdot$2H$_2$O</td>
<td>Molecular weight 172.18</td>
</tr>
<tr>
<td>Gypsum 0.2 g/L</td>
<td>0.61%</td>
</tr>
<tr>
<td>0.3 g/L</td>
<td>0.91</td>
</tr>
<tr>
<td>Sodium sulfate decahydrate Mirabilite Na$_2$SO$_4 \cdot$10H$_2$O</td>
<td>322.22</td>
</tr>
<tr>
<td>Mirabilite</td>
<td>1.14%</td>
</tr>
</tbody>
</table>
### Durham Sandstone Unweathered

#### Water Absorption/Drying Behavior Data

<table>
<thead>
<tr>
<th>Time Elapsed (min)</th>
<th>Cumulative Time (sec)</th>
<th>Sq. Rt. Of Time (sec^{0.5})</th>
<th>Weight of Sample - Wt (g)</th>
<th>Amount of Water Absorbed - Ut (g)</th>
<th>Amount of Water Absorbed per Unit Surface - M_i (g/cm^2)</th>
<th>Ut/W</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>0</td>
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**Initial Absorption Rate** 0.000187259
Durham Sandstone with Alveolar Erosion

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**Imbibition Capacity:**
\[
\frac{(W_{\text{max}} - W_{\text{dry}})}{W_{\text{dry}}} = \frac{430.24 - 407.01}{407.01} = 0.06
\]

**% Apparent Porosity:**
\[
\frac{(W_{\text{max}} - W_{\text{dry}})}{100} = \frac{430.24 - 407.01}{100} = 5.71
\]

**% Open Porosity:**
\[
\frac{W_{\text{op}}}{100} = \frac{23.23}{100} = 23.23 \text{ cm}^3
\]

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Imbibition Capacity: 
\[
\frac{W_{\text{max}} - W_{\text{dry}}}{W_{\text{dry}}} = \frac{416.13 - 393.98}{393.98} = 0.06
\]

% Apparent Porosity: 
\[
\frac{W_{\text{max}} - W_{\text{dry}}}{100 / W_{\text{dry}}} = \frac{417.13 - 393.98}{100 / 393.98} = 5.88
\]

% Open Porosity = \(\left(\frac{\text{Vop} \times 100}{125}\right)\) = \(\frac{23.15\text{g}}{1\text{g/cm}^3} \times \frac{100}{125}\) = 18.52
### Stone A - Center Area 1 Report (Flat)

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### Stone A - Edge Area 1 Report

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F.15
appendix g. laboratory exercises and data sheets.
Ex. 21 MORTAR ANALYSIS: SIMPLE METHOD

INTRODUCTION/AIM

The following test is a simple method for determining the proportions of the three principal components of an historic mortar: (1) the binder (basically calcium carbonate (CaCO₃), soluble in acid); (2) the fines (finely-textured impurities such as clays); and (3) the sand or aggregate. The analysis gives approximate information and should be carried out together with other analyses (i.e., mineralogical, microscopic, etc.) in order to derive the maximum possible information from the mortar sample, either for the purposes of historical research or to prepare a restoration mortar compatible with the original.

As regards the latter, it should be remembered that several important factors that affect the condition and performance of a mortar are not revealed in mortar analysis. These include the original water:binder ratio, the mixing and placing method, the rate of drying, and the cleanliness and condition of the aggregates. In some ways, the most useful aspect of mortar analysis is the identification of aggregates for matching.

EQUIPMENT/CHEMICALS

Oven, balance, mortar and pestle, beakers, funnel, wash bottle, filter paper, glass rods, heat lamp, sieve set, 14% solution of hydrochloric acid, deionized water.

PROCEDURE

A. Collection/Examination/Dissolution of Binder

1. Collect a sample of adequate size (about 40-50 grams).

2. Examine the sample and record the following characteristics: color, texture, inclusions, hardness.

3. Powder half of the sample (20-25 grams) with a mortar and pestle; leave the other half for further analysis.

4. Dry the powdered sample in the oven at 110°C for 24 hours and then weigh it (W₁) with a balance (0.01 g precision). Record the weight on the attached data sheet.

5. Place the sample in a 600 mL beaker and moisten it with water.

6. Immerse the moistened sample in a 14% solution of hydrochloric acid in order to dissolve the binder.

Observe the reaction and record observations.
Ex. 21 (continued)

B. Separation/ Filtration/ Sieving

1. Label the filter paper to be used, weigh it \( W_2 \), and record the weight on the data sheet.

2. Fold the paper into quarters and place it in a funnel. Position the funnel so that it will drain into a large flask.

3. Add a few drops of hydrochloric acid (HCl) to the sample to verify complete acid digestion of the binder (i.e. that the reaction initiated in Step 6 above is complete).

4. Slowly add water to the remaining sample material.

5. Swirl with a glass rod to suspend the fines.

6. Slowly pour the liquid with suspended material through the filter, being careful to keep solid particles (sand) at the bottom of the beaker.

7. Repeat process (steps 4–6) until the water added to the beaker remains clear.

8. Dry the fines collected on the filter paper with a heat lamp.

9. Wash the sand with water several times and leave it to dry for 24 hours.

10. Weigh the filter paper with the dry fines \( W_3 \). Subtract the weight of the paper \( W_2 \) to determine the weight of the fines \( W_3 - W_2 \). Record both values on the data sheet.

11. Weigh the dry sand \( W_4 \) and record the weight on the data sheet.

12. Express the amount of sand as a w/w percentage of the whole sample. Express the amount of fines in the same manner. The amount of dissolved binder is calculated by summing up the percentages of sand and fines and subtracting from 100%.

C. Characterization of Sand

1. Examine the sand with a binocular microscope. Record characteristics: color, particle shape and size, etc.
Ex. 21 (continued)

2. Sieve the sand in a standard sieve set to determine the particle size distribution. Express the amount of each particle size as a percentage of the whole (see Ex. 18A).

* The limitations of mortar analysis should always be considered. For example, calcareous aggregates will be dissolved with calcareous binder material and present misleading binder-aggregate proportions. Similarly, clay minerals present as impurities may not be readily distinguishable from the silicates present in hydraulic cement (see Ashurst, p. 20).

BIBLIOGRAPHY


Columbia University, Historic Preservation Program. Laboratory exercises.

HSPV-656  Advanced Architectural Conservation

Laboratory Notes  Prof. A. Elena Charola

Determination of moisture and soluble salt content in a sample

Moisture content

The solid sample is weighed on a watch-glass or appropriate sample holder. For this purpose:

- weigh the sample holder by itself (w_a)
- weigh the sample on the sample holder (w_{a,s})

Calculate the weight of the sample

\[ w_s = (w_{a,s} - w_a) \]

Put the sample on its sample holder to dry in an oven at 105°C. Leave it for at least 24 hours. Take the sample, on its sample holder, out of the oven and put it into a desiccator to cool down to room temperature. (Make sure the desiccant is not saturated.) Weigh the sample.

Return the sample to the oven for at least another 2 hours and repeat the above procedure until the difference in weight of two consecutive weighings is less than 0.01% of the weight of the sample. This is \( w_{a,d} \).

Calculate the weight of the dry sample

\[ w_d = (w_{a,d} - w_a) \]

Calculate the % moisture content in the sample:

\[
\text{% Moisture content (w/w)} = \frac{w_s - w_d}{w_d} \times 100
\]

Soluble salt content

Grind the dry sample in an agate mortar until a uniform coarse powder is obtained or the sample is broken down into individual grains. Weigh the sample following the procedure described above (make sure you weigh the holder by itself first). Calculate the weight of your sample \((w'_s)\). Dry the sample for at least 2 hours and weigh it, repeating the heating process until constant weight. Calculate the dry weight of sample \((w'_{d})\). Even thought the sample has been dried in advance of the crushing, the difference between \(w'_s\) and \(w'_{d}\) is the moisture picked up during grinding and handling.

Put the sample into an appropriately sized beaker and add sufficient distilled water. Add a magnetic stirring bar and put the beaker on magnetic stirrer. Leave stirring for 2 hours. Take the stirrer out and leave the suspension to settle overnight.*

* The purpose of the magnetic stirrer is to insure that soluble salts in the sample are completely dissolved in the water to form a solution. If your sample contains hard, abrasive inclusions, such as sand grains in brick,
Filter the suspension through a previously weighed filter paper \((w_p)\) (preferably ashless filter paper S&S 589 blue or black ribbon) into another beaker of appropriate size, making sure to transfer all solid residue into the filter paper with distilled water.

Measure the volume of the solution obtained (if necessary, transfer into a graduated cylinder). Make a note of the final volume \((V_{\text{soln}})\) of this solution and save it for the identification of soluble salts and their concentration. Accurate measurement of this final volume is more important than the amount of water you added initially.

Pre-weight a watch-glass \((w_d)\) and transfer the solid with the filter paper onto it \((w_{d\text{-wet}})\), then put it to dry in the oven for 24 hours. Take the sample, on its sample holder, out of the oven and put it into a desiccator to cool down to room temperature. Weigh the sample. Return the sample to the oven leaving it for at least 2 hours and repeat the above procedure until the differences in weight of two consecutive weighings is less than 0.01\% of the weight of the sample. This is \((w_{d\text{-dry}})\). Calculate the dry weight of the sample, including the accruing uncertainties.

\[
w_{d\text{yr}} = (w_{d\text{-dry}}) - (w_d) ± 2\Delta w
\]

Calculate the \% weight of soluble salt content in the sample:

\[
\% \text{ Soluble Salt content (w/w)} = \frac{(w_d' - w_{d\text{yr}} - w_p)}{w_d} \times 100
\]

You have just completed one method of calculating the soluble salt content (by weight) in your sample. This method allows one to find the weight of the soluble material without finding the specific type of salt. Another method of finding soluble salt content, one that identifies salt content by type is described below.

**Semiquantitative analysis of soluble salts present in the sample**

From the solution created above, aliquots are taken for the identification of the ions present by semi-micro chemical reactions or spot tests, or by identification with commercial strips. The strips are then used for a quantitative analysis of ion content; this analysis is sometimes called “semi-quantitative” because the strips only provide data within a certain range and thus are not extremely precise.

From this point, follow the directions in the Laboratory Notes *Ion Test Strips for Semiquantitative Concentration Determination* in order to determine the original salt quantities (by salt type) present in the sample.

---

they will erode the teflon coating of the magnetic bar thus exposing its iron core. In this case, reduce the time of stirring to half an hour. Leave for an hour, stir again for half an hour, and then leave to decant as above. Or use a glass stirring rod. Stir for five minutes, wait fifteen minutes, and repeat several times. Then allow the suspension to settle and continue as above.
Objective:
Correct use of commercial ion test strips.
Calculation of the approximate concentration of the ion in a given sample.

Commercially available test strips for different ions operate similarly to the pH strips. They are very practical since apart from identifying the presence of the ion in question they can also provide a semiquantitative value of the concentration of the ion in the particular solution.

Experimental:
Commercial strips are available for various ions, such as Cl\(^-\), SO\(_4\)^{2-}, NO\(_3\)^{-}, NO\(_2\)^{-}, PO\(_4\)^{3-} and NH\(_4\)^{+}. Note that the concentration range within which an ion can be measured depends on the ion and the particular brand of the strip. One of the drawbacks of these test strips is their cost. Therefore, their use for ion identification purposes should be limited for in-situ testing, while in the laboratory, they should ONLY be used for the semiquantitative determination of a given ion in a given sample. Samples in architectural preservation are usually solids: either an efflorescence or a porous material that is contaminated with salt.

1. Identification of the ion(s) present

Once a sample is obtained, the first step is to determine the presence of the contamination ions via micro spot tests. For this purpose, a little amount of sample is only necessary (if it is an efflorescence or in a powder form). If the salt is within a porous material, it needs to be extracted (see Lab Notes for Determination of Moisture and Soluble Salt Content).

2. Semiquantitative determination of the ion(s)

Enough sample must be available for weighing, this means that at least 0.5 g of the sample is necessary for a nearly pure efflorescence, or 1 g if it also contains powder of the deteriorating material.

The weighed sample—remember to subtract the weight of the container—is put into a small beaker and dissolved in water (if you have a powdered sample taken from the surface of a deteriorating stone/render/brick, only the salt will go into solution and there will be a residue. In this case, the powdered sample should be left in water for at least an hour with occasional stirring).

This solution is then taken to a given volume, e.g., 10 ml, 50 ml, etc., either in a graduated cylinder or in a volumetric flask, depending on the precision required. Record this volume which contains all the ions of your sample.
An aliquot of this solution is taken in a little beaker and the test strip immersed, or drops of the solution are put onto the strip (read the instructions for each type of test strip). Once the color has developed, the concentration of the ion in question is given by the test strip. Please note that some test strips give the concentration in the ion itself, i.e., NO$_3^-$, while others give it as a compound, i.e., NaCl. Also note that some may give the concentration in mg/l (ppm) or in g/l.

The concentration of the ion in the sample is then calculated as follows:

$$\text{Ion (g/g) } \% = \frac{\text{Strip Reading (mg/l)} \times V_{\text{soln}} (l) \times 100}{w_{\text{sample}} (g) \times 1000 \text{ mg/g}}$$

If the concentration of the ion in question is too high, a dilution must be prepared from the solution and this has to be taken into account in the calculation. Note that in this case the volume of the aliquot has to be measured exactly as well as the volume of the dilute solution. And then an unmeasured aliquot of this dilution is taken to make the measurement.

$$\text{Ion (g/g) } = \frac{\text{Strip Reading (mg/l)} \times V_{\text{soln}} (l) \times V_{\text{dil}} (ml)}{V_{\text{aliquot}} (ml) \times w_{\text{sample}} (g) \times 1000 \text{ mg/g}}$$

**Note:**

Technically, an aliquot means a part of a number or quantity that will divide it without a remainder; thus, 5 is an aliquot part of 15. In general, it means a measured smaller volume of a larger volume. For use with the strip, it does not have to be a measured aliquot, but if a dilution has to be prepared, then the volume of the aliquot needs to be known exactly as well as the volume of the original solution and of the dilution.

**Report:**

1. The type of sample you are analyzing, its appearance and size.
2. The ions identified in your sample and the microtests used.
3. The concentration of the solution prepared (w/v).
4. The reading of the test strip(s). The % concentration of the ion.
5. If dilutions had to be prepared, include the corresponding data: volume of aliquot, volume of dilution.
6. Reading of the test strip(s).
7. Identify in each case, the test strips used (brand and sensitivity range).
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Laboratory Notes

Water Absorption and Drying Behavior

Objective:  Determination of:

- Capillary water absorption curve & capillary water absorption coefficient.
- Water absorption by total immersion & porosity.
- Drying curve, drying rate and critical moisture content.
- Total immersion water absorption curve. Determination of the water capacity of the material.

Outline:

The determination of these parameters should be carried out in preference on regular shaped samples, such as 5x5x5 cm cubes, and in a continuous sequence:

I. The capillary water absorption is determined. Once the sample has reached the asymptotical water absorption value continue with point 2.

II. The sample is totally immersed in water and the total amount of water absorbed determined. See following section for further information.

III. The sample is then left to dry and the drying curve determined.


Procedure to be followed:

I. Capillary water absorption curve & capillary water absorption coefficient.

1. Measure the weight of the oven-dried (60°C) sample (W\text{dry}[^g]). Rest the dry sample on glass beads in a container. Fill the container with deionized water until the water level reaches the bottom of the sample and taking care that it is not so high as to touch the sides of the sample. Cover the container as hermetically as possible.

2. The sample needs to be weighed at different times as it absorbs water. This needs to be adjusted to the porosity of the sample. As a rule of thumb, readings have to be taken closely together at the beginning, and then spaced out as absorption decreases. As a rule of thumb, do a first run taking weighings every 5 minutes for the first half hour, then every 15 minutes for the second half hour. Then every half hour for the next two hours, and after that every hour until the water absorption stabilizes.
3. To weigh the sample, take it out, pat the bottom of the sample dry with a paper towel and put it on the balance. Take the reading as fast as possible and return the sample to the container, making sure to close it again.

4. After the sample has reached the asymptotical water absorption value, leave the sample 24 hours before taking a last weighing.

**Data presentation:**

- The data should be presented in a Table with at least six columns indicating: actual time of measurement, cumulative time [seconds], square root of time [sec$^{0.5}$], weight of sample $W_i$ [g], amount of water absorbed $U_i = (W_i - W_{dry})$ [g] and amount of water absorbed per unit surface $M_i$ (g/cm$^2$).
- The capillary water absorption curves plots $M_i$ vs. square root of time.
- The **Capillary Absorption Coefficient** is the slope of the initial straight part of the curve expressed in [g/cm$^2$ sec$^{0.5}$]. Also express the coefficient as [kg/m$^2$.h$^{0.5}$]. Make sure you also calculate the correlation factor (or coefficient) which is 1 for a straight line. This factor should not be lower than 0.995.

**II. Water absorption by total immersion and determination of porosity.**

1. After the last weighing the sample is immediately totally immersed in deionized water and left immersed for another 24 hours.

2. The sample is taken out of the water, lightly patted dry and weighed.

**Data presentation:**

- The weight of the sample after 24-h immersion is $W_{max}$ [g] and from it, the total water content, $U_n = (W_{max} - W_{dry})$ [g] is calculated.
- The **Inhibition Capacity** IC is the ratio $[W_{max} - W_{dry}] / W_{dry}$
- The % **Absorption; % Apparent Porosity** or the **Water Absorption Capacity** WAC is the percentange of the above value, that is $[W_{max} - W_{dry}] 100 / W_{dry}$
- The % **Open Porosity** is calculated based on the ratio of volume of open pores, $V_{op}$ [cm$^3$] to the total volume of the sample, $Vs$ [cm$^3$]. The volume of open pores $V_{op}$ [cm$^3$] is estimated from the total water content $U_0$ assuming that the density of water is practically 1 g/cm$^3$. The volume of the sample, $Vs$ [cm$^3$] can be calculated from its geometrical shape, i.e., 125 cm$^3$ for a 5x5x5cm cube.

\[
\% \text{ Open Porosity} = \left( V_{op} \times 100 \right) / 125 \]
III. Drying Curve

1. As soon as the sample is taken out of the water to be weighed in the previous step, it is best left to dry on the balance for the first half hour, taking readings every 2 or 3 minutes (depending on the porosity of the sample) during the first 15 minutes and then every five minutes.

2. Once the weight of the sample does not change so fast, it can be put to dry on a rack in a draught free environment and its weight loss measured. As a rule of thumb, after the first half hour, weighing should be made every ten minutes for another hour, and then every fifteen minutes, then half an hour, etc.

3. After the weight has reached an asymptotical value, the sample is dried again in the oven, and this value is used as the dry weight $[W_d]$ for the calculations. If this value changed from the original one, recalculate the total water content $U_o$ using this value.

**Data presentation:**

- The data is presented in a table with at least seven columns indicating: actual time of measurement; cumulative time [hours]; weight of sample $[W_i]$, water content $U_i$ [g]; relative moisture content $Y$ ($Y = U_i/U_o$); residual water content $Q%$ ($U_i \times 100/W_d$); and, moisture content $\Psi$ ($\Psi = (U_i/Vol_{sample})$ (g/cm$^3$).
- The drying curves are plotted as: $Q%$ vs. time; $\Psi$ vs. time; and, $Y$ vs. time.
- Use the $\Psi$ vs. time graph to plot the drying rate curve. For this purpose, you need to identify up to which time point the curve is actually a straight line. This you can first estimate with a ruler and then calculate the correlation factor, which should not be lower than 0.995 for this part of the curve. Then you calculate the slope of the curve. This gives you the initial drying rate.
- The last point of the initial drying rate corresponds to the Critical Moisture Content ($\Psi_c$) and is expressed in g/cm$^3$ or kg/cm$^3$.
- Repeat this procedure for the flat end of the curve. In this case the correlation factor should not be lower than 0.995. Calculate the slope corresponding the that set of data. This is the final drying rate.
- The points in between are simple calculated as follows: $[(\Psi_c - \Psi_{t+1})/(t_{t+1}-t_t)]$ for time $(t_{t+1})$, and so forth until the first value of the final drying rate is reached.
- Construct a table where you use the initial drying rate up to time $t_c$, then use the above calculated points up to the first point which was used in the calculation of the final drying rate, and use the final drying rate for the rest of the time points. Make sure you change the sign for the drying rate.
- Plot the drying rate as a function of time (hours) using the “line type” chart in Excel. Note, only drying rate column needs to be highlighted. The abscissa (x axis) is automatically put in by the program. Make sure you eliminate the numbers introduced by Excel for this axis since they only represent the “reading” events.
Technical Information Sheet

Funcosil®
Antihygro

Art.No. 8616

Preservative that stops swelling in natural stone which has clayey binders to reduce hygroscopic swelling.

Product lines:

Anti-hygro concretes, mortars, etc.

Property profile:

Funcosil Antihygro clearly differs in the way it works from other stone preservatives. This is an agent that reduces the hygroscopic swelling value of natural stone by approx. 50% without considerably influencing water absorption and physical- mechanical behaviour of the stone. The effect of Funcosil Antihygro is based on inhibition of swelling capable centres in the stone (silica gel playa marina). According to the knowledge we have today, swelling and shrinking processes as the result of moisture penetration and drying are seen as the primary cause of damage in clayey sandstone, but also to some extent in brick and full stone material. These swelling processes can occur at average to high humidity. Pretreatment with Funcosil Antihygro, especially for highly swelling capable, clayey sandstone that has little resistance to swelling because of hygroscopic swelling, is an imperative.

Characteristic data of the product in the packaged state:

Effective ingredient content: 0.2 vol%
Density at 20°C: approx. 1.01 kg/l
pH value: 6 ± 1
Solvent: water
Colour: clear

Range of use:

To be used for all stones, clays, cements, building materials with silicate in high swelling value.

Substrate conditions/substrate pre-treatment:

A prerequisite for an optimal effect with Funcosil Antihygro is optimal penetration of the effective ingredients. In order to achieve this, the following points should be observed:

- The surface in to be treated may not have been hydrophobically treated (water repelling); for this reason, a

CIS 8010 91.81 ston

Preservatives — protect the value of buildings.
Technical Information Sheet

Follow-up treatment:
Natural stone preservation can be executed in the Fencosil System with a manufacturer package co-ordinated to the damage and weathering mechanism. To support the effect of Fencosil Antikopp, it is advisable in many cases to use a stone strengtheners (Fencosil Stone Strengtheners 90°) and in some cases Fencosil Stone Strengtheners 100 or Fencosil Stone Strengtheners 50° and/or a hydrophobic impregnation (Fencosil ST, SBNL, MSIL). Coloured coating of the surface can be carried out with the Fencosil Silicone Emulsion Paint System. Working details and specifications of the products named are found in the respective Technical Information Sheets.

Tools

All non-corrosive, low pressure carriers or spraying equipment, liquid guns and especially the Fencosil MSIL Sprayer are suitable for working.

Packaging, application rate and storing:

Packaging: 5 l and 30 l plastic containers

Application rate:
Rack treatment: 10-15 l/m²
Method treatment: 0.3-0.5 l/m²
Tuff: 10-15 l/m²
Back: 10-15 l/m²
The exact requirements should be determined on a sufficiently large (1.2 m²) test surface.

Shelf life: At least 6 months in closed, mineral containers, stored cool and frost-free. Fencosil Antikopp reacts with oxygen, so close containers air-tight each time material is removed. Protect containers from strong sunlight

Safety, ecology, disposal:

Further information concerning safety during transport, storage and handling as well as the disposal is found in the latest Safety Data Sheet.

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In all cases, our general conditions of sale are valid.

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Remmers KSE 300 E is preferably used for friable, medium to coarse-pored sandstone, certain volcanic rock (e.g. tuff) as well as weathered brick. It can also be used for strengthening historical renders and joints. Stone that has pronounced swelling and shrinking properties due to swelling capable clay minerals must be treated first with Funcosil Antihygro (Art. No. 0816) to reduce swelling. The stone should be examined in Remmers’ laboratory.

Property profile
Remmers KSE 300 E, an elastified stone strenghtener, was developed in co-operation with Dr. E. Wendler (Munich) and a work group directed by Prof. Dr. J. Grobe (Münster) within the framework of a project called "Protecting Stone Surfaces through the Application of Elastic Silicic Acid Ester" which was sponsored by the German Federal Environment Foundation (Os nabrück). Remmers KSE 300 E differs from conventional stone strengtheners by a

- moderate E-modulus increase (stress-strain behaviour) while
- at the same time providing

- sufficient consolidation of the natural stone structure.

Remmers KSE 300 E reacts with the water or humidity stored in the pore system. During this reaction, amorphous and hydrous silicon dioxide linked through soft segments is deposited as a binder. The binder silca gel replaces the original binder lost through weathering.

The speed of the gel deposit reaction is highly dependent on temperature and humidity. Under normal conditions (20°C, 50% relative humidity), the deposit of binder is concluded after approx. three weeks.

The most important property parameters of Remmers KSE 300 E stone strenghtener are listed in the following:

- Gel deposit rate approx. 30 %
- Single component system – reliable and easy to use
- Neutral catalyst
- High penetration depth, possible all the way down to the sound core of the stone material
- No by-products that damage the building
- High weather resistance and UV stability
- Partially strengthened natural stone can be worked over with Remmers Restoration Mortar.

Directions
Preliminary examination, setting up trial areas.

The following characteristic properties of the material should be
determined (analysis of the state of the building):
1. Moisture content, content of damaging salts, hygroscopic water absorption
2. Absorptivity, capillary water absorption
3. Strength profile, depth of weathering, degree of hygroscopic swelling
4. Application rate for each area, penetration depth of the stone strengthening, resulting strength profile
5. Establishment of working operations
6. Set-up of a representative trial area which is necessary to see if there will be changes in colour and the correlation between laboratory results and the quantities and values achieved on the object.
7. Execution of treatment and application rates should be controlled and documented.

Preparing the substrate:
Surfaces to be restored often show reduced absorption capacity due to a crust of soil or different types of “patina”. The cleaning measures necessary to restore the original absorption behaviour should be as gentle as possible, e.g. by spraying with cold or warm water or by steam cleaning. In case of stubborn soil, the Rotec Low Pressure Blasting Device should be preferentially used or Remmers cleaning products (see the respective Technical Information Sheets).

In many cases the stone is already so friable that cleaning cannot take place without a sensitive loss of substance. To avoid this, pre-strengthening with Remmers KSE 300 E or another suitable stone strengthening from the Remmers KSE family can be carried out prior to cleaning. The main strengthening measure is then carried out after the cleaned surface has dried.
In order to achieve complete saturation of the weathered zone of the stone with Remmers KSE 300 E, the surface to be treated must have reached compensation moisture balance, be absorbent and should not have been heated by the sun. When strengthening is carried out, the temperature of the stone strengthening as well as the temperature of the substrate and surrounding air should range between 8 °C and 25 °C. To avoid strong heating, use shading devices. The surfaces should be protected from sun, rain and wind before, during and after strengthening.

Application procedures:
An essential prerequisite for optimal strengthening is that the weathered zone is completely saturated all the way down to the sound core. To achieve this, Remmers KSE 300 E stone strengthening is applied to the building material in a flow coating, dipping and/or compress procedure. When using a flow coating procedure, smaller areas (if necessary, stone by stone) are treated, wet-on-wet, with KSE 300 E until the material is no longer absorbed. The procedure selected for application always depends on the task at hand. So-called “fast hydrolysis” is not recommended since this represents an uncontrolled influence on the gel formation reaction and therefore the success of the strengthening measure.

Notes
If necessary, treatment can be repeated 2-3 weeks after initial treatment. In this case as well, saturation of the complete weathered zone must be achieved. The application rate of Remmers KSE 300 E should be determined in the laboratory during preliminary examinations and on a trial surface and will depend not only on the absorptivity of the substrate but also on the application procedure selected.

Follow up treatment:
To avoid a change in the colour of the surface caused by over-saturation with Remmers KSE 300 E, the stone surface should be washed off with a dry solvent (e.g. Thinner V 101) immediately after saturation has been achieved.

Application of stone substitution compounds, hydrophobizing impregnation agents and coats of paint:
Surfaces that have been strengthened with Remmers KSE 300 E can be worked over – after the deposit of gel has been concluded – with Remmers Restoration Mortar, Funcosil impregnation agents and/or products in the Remmers Silicone Resin Paint System. After application, the “siliceic acid ester” chemical system leads to a temporary water repelling effect which disappears during the course of gel formation. If strengthened surfaces still show an annoying water repelling effect when restoration mortar is subsequently applied, this can be suppressed by wetting the surface with alcohol.

Adjoining surfaces:
Facade elements that should not come in contact with the impregnation agent such as, e.g. windows, varnished surfaces, glass and also plants should be protected by suitable measures (e.g. covered with plastic sheets).

Tools, cleaning
Depending on the task at hand, e.g. low pressure spraying equipment, airless equipment, hand sprayers. Tools and equipment must be clean and dry. After use and before longer pauses, they should be cleaned thoroughly with Thinner V 101. Once the stone strengthening has reacted, it can only be removed mechanically.

Packaging, application rate, shelf-life
Packaging:
5 l, 30 l and 200 l tin containers
Application rate:
The application rate of Remmers KSE 300 E depends to a considerable degree on the type and state of the substrate to be treated as well as the task at hand and the application technique used. The quantity required may vary ac-
cordingly and can range between 0.1 l/m² to several litres per m².

The rate should be determined in the laboratory during preliminary examination as well as on a trial surface.

**Safety, ecology, disposal**

Further information on safety when transporting, storing and handling as well as disposal and ecology is found in the latest Safety Data Sheet.

**Personal protective equipment** is required for spraying procedures. Use respiratory protection with a combination filter at least A/P2 (made by e.g. Dräger). For suitable protective gloves, see Safety Data Sheet. Wear closed work clothes.

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Of all materials currently and historically employed in construction, masonry is one of the most durable. What has become apparent in recent years, however, is that masonry materials are not as enduring as once believed. Placed in contemporary urban environments, these “timeless” materials decay at an alarming rate. Some deterioration may be attributed to the masonry’s natural weathering process. The majority of the deterioration, however, is the result of oversights in use and maintenance of the masonry, and of the impact that industrialization has had on our environment, i.e. “acid deposition.”

The intent of all conservation treatments is to restore the structural integrity to crumbling, decaying masonry and/or provide a means of controlling future decay. The failure of many conservation treatments lies in their inherent dissimilarity to the masonry for which they are proposed as a preservative. When selecting a conservation treatment, an important consideration is to identify those treatments with physical and chemical characteristics similar to the masonry itself.

Conservare® Consolidation Treatments are based on silicic ethyl esters. Their extremely small molecular structure enables them to penetrate deeply into deteriorated masonry surfaces, collecting at contact points between individual stone grains. An internal catalyst and atmospheric humidity then convert the liquid consolidant into a glass-like silicon dioxide (SiO₂) gel which binds the stone particles together. Exhibiting chemical characteristics and thermal expansion/contraction characteristics which are virtually identical to that of natural stone, the newly deposited SiO₂ cementing matrix replaces the stone’s natural cement which has been lost due to weathering influences.

**DESCRIPTION AND USE**

Conservare® OH100 is a ready-to-use consolidation treatment that stabilizes masonry by replacing the natural binding materials, lost due to weathering, with silicon dioxide. When properly applied, Conservare® OH100 penetrates deeply, does not form a dense surface crust, and retains the substrate’s natural vapor permeability. In addition to the general consolidation of severely deteriorated masonries, Conservare® OH100 is an effective pretreatment for friable substrates that need to be strengthened before cleaning, patching or coating. Conservare® OH100 may be used on most types of natural stone, concrete, stucco, brick, terra-cotta, etc.

Conservare® OH100 is effective on unpolished marble, travertine and limestone that has been treated with Conservare® HCT (Hydroxylating Conversion Treatment).

**ADVANTAGES**

- One component – easy-to-use. Strengthens deteriorated stone.
- Low viscosity allows deep penetration. Will not form hardened surface crust.
- The new binder is mineral – similar to the original stone – no synthetic polymers.
- Rapid tack free drying – no dirt attraction.
- Forms no by-products harmful to the masonry.
- Treated surfaces “breathe” – does not trap moisture.
- New binder is acid resistant – resists acid rain.

**Limitations**

- Effective consolidation requires thorough laboratory and field pretesting. Contact PROSOCO for information on the recommended test programs.
- Limited shelf life – remains storage stable for approximately 12 months in sealed containers. Treated areas may bond to silicone and polyurethane molds (frequently used for casting replacement stone). Use a release agent to prevent molding compounds from adhering to the treated surface.
- Not suitable for some types of marble.

### OH100 Consolidation Treatment

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Type</th>
<th>Use?</th>
<th>Coverage*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Architectural Concrete Block</td>
<td>Burnished</td>
<td>no</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td>Smooth</td>
<td>no</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ribbed</td>
<td>no</td>
<td></td>
</tr>
<tr>
<td>Concrete</td>
<td>Brick</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
<tr>
<td></td>
<td>Tile</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Precast Panels</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pavers</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Cast-in-place</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td>Fired Clay</td>
<td>Brick</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
<tr>
<td></td>
<td>Tile</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Terra Cotta</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Pavers</td>
<td>yes</td>
<td></td>
</tr>
<tr>
<td>Marble, Travertine, Limestone</td>
<td>Polished</td>
<td>no</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td>Unpolished</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
<tr>
<td>Granite</td>
<td>Polished</td>
<td>no</td>
<td>NA</td>
</tr>
<tr>
<td></td>
<td>Unpolished</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
<tr>
<td>Sandstone</td>
<td>Unpolished</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
<tr>
<td>Slate</td>
<td>Unpolished</td>
<td>yes</td>
<td>Determine via on-site test panels.</td>
</tr>
</tbody>
</table>

*Laboratory and field testing are necessary to confirm desired results and application procedures. Coverage rates vary, depending on degree of deterioration and recommended application procedures.

**PROSOCO OH100**

Conservare® OH100 Consolidation Treatment

penetrating stone & masonry strengthener
TYPICAL TECHNICAL DATA
FORM: Colorless to slight yellow.
SPECIFIC GRAVITY: 0.997
pH: NA
WT/GAL: 8.30 lbs.
ACTIVE CONTENT: 100%
TOTAL SOLIDS: 43% ASTM D 5095
FLASH POINT: 104°F (40°C)
FREEZE POINT: -22°F (-30°C)
SHELF LIFE: 1 year in a tightly sealed, unopened container

VOC Information

PREPARATION
Protect people, vehicles, property, metal, glass, foliage, painted surfaces and all nonmasonry surfaces from contact with product, fumes or wind drift. Protect and/or divert pedestrian and auto traffic. Ensure fresh air entry and cross ventilation during application and drying. Extinguish all flames, pilot lights and other potential sources of ignition during use and until all vapors are gone. When applying to exteriors of occupied buildings, make sure all windows, exterior intakes and air conditioning vents are covered and air handling equipment is shut down during application and until all vapors have dissipated.

The Importance of Pretesting
Since building materials differ in their nature and degree of deterioration, each conservation project poses unique problems and requirements. To gain a full understanding of the ongoing deterioration and determine necessary stabilization/conservation measures, a number of laboratory and field tests are required.

a. Evaluates the physical and chemical characteristics of the substrate(s) to confirm whether consolidation is possible.
b. Identifies the cause(s) of deterioration and surface preparation procedures necessary for conservation treatment.
c. Determines the most appropriate conservation agent(s) and field application procedures.

For more information on the recommended testing program, read the Conservare® Stone Testing Brochure and contact your PROSOCO representative to arrange a job-site visit.

On-Site Testing: Following lab testing, a test area should be cleaned and allowed to dry. An application of Conservare® OH100 Consolidation Treatment is made following specific recommendations provided by the laboratory analysis. The job site test area should be as large as possible and representative of the condition of the entire project. The test area is necessary to confirm application procedures under job site conditions and allow calculation of the masonry’s consumption rate. The on-site tests also provide a visible sample of the effects of the treatment on actual job surfaces. Additional core samples can be taken from the test area and tested to verify depth of penetration and proper application procedures.

Surface Preparation
Following lab and on-site testing, clean the building with the appropriate Sure Klean® product. In most cases, surface contaminants such as carbon crust, salts, pigeon droppings, moldew and atmospheric stains must be completely removed to assure thorough penetration of Conservare® OH100. In addition, surface sealers and repellents which may have been applied must be thoroughly removed. Contact Customer Care at (800) 255-4255 for additional cleaning recommendations.

*In cases where even the most sympathetic cleaning program would remove an unacceptable level of surface detail, Conservare® OH100 Consolidation Treatment may be applied to the soiled surface to preconsolidate the stone. If such pre-consolidation is necessary, further evaluation will be required to ensure that no undesirable reactions take place between the consolidation treatment and the surface contaminants which may interfere with further conservation measures, i.e. subsequent cleaning, general consolidation, patching/repair, etc.

Surface and Air Temperatures
Protect surface to be treated from direct sunlight for several hours prior to beginning application. When possible, initiate treatment when surfaces are shaded. Keep surface temperature relatively cool to prevent too rapid evaporation of Conservare® OH100 and to ensure proper penetration. Do not apply during rain, to wet surfaces or when there is a chance of rain. Protect from rain for two days following application. Surface and air temperatures should be between 50-90°F (10-32°C) during application. Relative humidity should be greater than 40%. Excessive surface heating can be prevented by shading with awnings.

Storage and Handling
Store in a cool, dry place away from potential ignition sources. Keep tightly closed when not dispensing. Published shelf life assumes upright storage of factory-sealed containers in a dry place. Maintain temperature of 45-100°F (7-38°C). Do not double stack pallets. Dispose of unused product and container in accordance with local, state and federal regulations.

APPLICATION
Before use, read “Preparation” and “Safety Information.”

Dilutions
Use in concentrate. Do not dilute or alter. Stir or mix well before use.

Application Instructions
Apply by low-pressure spray, brush or dipping. Larger surfaces should be treated using low-pressure spray equipment, small areas with spray tanks. Mobile objects such as sculptures are best treated indoors by dipping or with the use of compresses. Contact
Ensure proper penetration and prevent crust formations by applying Conservare® OH100 in repeated applications referred to as “cycles.” A cycle consists of three successive saturating applications at 5-15 minute intervals.

Typical treatments involve two or three cycles (6-9 separate applications). Allow 20 to 60 minutes between cycles. Laboratory testing will determine the optimum delay between applications and between cycles. Additional material should be applied until excess material remains visible on the surface for 60 minutes following the last application. Once this degree of saturation is achieved over the entire surface, the first treatment is complete. Immediately flush excess surface materials using industrial grade MEK (methyl ethyl ketone) or mineral spirits. If a second treatment is necessary, allow two to three hours curing time following first treatment.

Proper timing of the application process will maximize penetration of the consolidation treatment. Deep penetration is critical to the long-term benefits of any consolidation treatment.

Cleanup
Clean tools and equipment immediately with mineral spirits, denatured alcohol or an equivalent cleaning solvent. Remove overspray and spills as soon as possible.

Post-Treatment
Areas properly treated with Conservare® OH100 can receive stone repair materials, regrouting materials and PROSOCO’s BMC® silicone emulsion paints after the consolidation procedures have been completed. After curing apply the appropriate Sure Klean® Weather Seal water repellent to ensure protection from further water damage.

SAFETY INFORMATION
Conservare® OH100 Consolidation Treatment is a solvent carried product and may cause symptoms typical with organic solvent exposures. This is a combustible material. Use appropriate ventilation, safety equipment and job site controls during application and handling. Read the full label for precautionary instructions before use.

First Aid
Ingestion: If swallowed, call a physician immediately. Do not induce vomiting except at the instruction of a physician. If vomiting occurs, keep head below waist to prevent entry of liquid into lungs.

Eye Contact: Rinse eyes thoroughly for 15 minutes. Get medical assistance.

Skin Contact: Rinse thoroughly. Get medical attention if irritation persists. Launder contaminated clothing before reuse.

Inhalation: Remove to fresh air. Give artificial respiration if not breathing. Get immediate medical attention.

24-Hour Emergency Information: INFOTRAC at 800-535-5053

WARRANTY
The information and recommendations made are based on our own research and the research of others, and are believed to be accurate. However, no guarantee of their accuracy is made because we cannot cover every possible application of our products, nor anticipate every variation encountered in masonry surfaces, job conditions and methods used. The purchasers shall make their own tests to determine the suitability of such products for a particular purpose. PROSOCO, Inc. warrants this product to be free from defects. Where permitted by law, PROSOCO makes no other warranties with respect to this product, express or implied, including without limitation the implied warranties of merchantability or fitness for particular purpose.

Customer Care
Factory personnel are available for product, environment and job safety assistance with no obligation. Call 800-255-4255 and ask for Customer Care. Factory-trained representatives are established in principal cities throughout the continental United States. Call Customer Care at 800-255-4255, or visit our web site at www.prosoco.com, for the name of the Conservare® representative in your area.
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