Historic England

Nanolime

A Practical Guide to its Use for Consolidating Weathered Limestone

Summary

This technical advice note is aimed at conservators and those specifying conservation treatments for historic stonework. It will also be of interest to conservation officers, and building owners and managers. Although the past ten years have seen an increase in the use of nanolime as a stone consolidant, not much was known about its properties and performance, and there had been no long-term evaluation of its effect on deteriorated limestone in an external UK environment. Furthermore, there was no consistent guidance regarding application of nanolime. For these reasons, Historic England commissioned a programme of research at the University of Bath and site trials at various English cathedrals. The information provided in this advice note is based on both the results of the research and the experience of conservators who have used nanolime. It also reflects issues discussed at a symposium held at the University of Bath in September 2015.

This information will aid practitioners and specifiers to make informed decisions about when and how to use nanolime. This document describes:

- the performance requirements and essential properties of consolidants in general
- the scientific theory underpinning the use of nanolime
- factors that might limit the effectiveness of nanolime
- the best ways to assess the suitability of stone for treatment with nanolime
- how to apply nanolime

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Front cover: Applying nanolime E5 to decayed areas of a limestone memorial in Kensal Green Cemetery.
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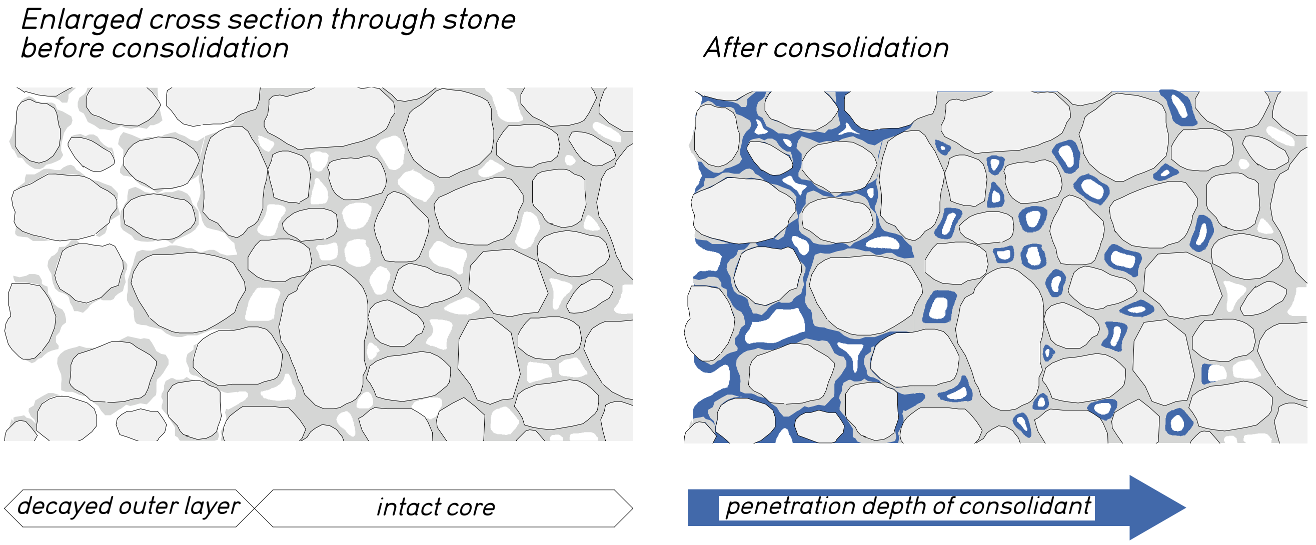
Introduction

Background

The decay of external limestone in England is a common phenomenon that has been acknowledged for many centuries. It is usually due to a combination of factors. These include the nature of the stone, the way in which it has been used, the environment in which it is set, the effects of moisture and pollution, and previous remedial treatments. Decay varies according to the type and properties of the stone and can be manifested in a number of ways. A common decay mechanism is dissolution and loss of binder (usually calcite), which reduces stone cohesion, leading to powdering and granular disintegration. The depth to which this is evident is usually between 1mm to 10mm, but in severe cases can be more.

There is a long history of attempts to consolidate decayed stone. Documentary records show that a range of materials have been used, including organic oils, natural resins, materials based on natural proteins (such as casein and albumen), limewater, silicate solutions such as ‘waterglass’ (sodium silicate), and ethyl silicate. It is likely that many of these materials consolidated or protected only the surface of the stone. However, if consolidants are to be effective in the long term, they must penetrate into sound stone below the zone of decay. In cases where deterioration is limited to a couple of millimetres, surface consolidation might be sufficient, but if the decay is deeper than that, the resulting interface between the consolidated surface and the decayed stone underneath can provide a potential zone for detachment.


Enlarged cross section through stone before consolidation

After consolidation

Figure 1
Diagram showing ideal consolidation in which the consolidant penetrates though the area of decay and into the sound core of the stone thus avoiding any interface between the treated and untreated stone.

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Properties and performance requirements of consolidants

Performance requirements

Current good practice amongst conservators has set a high bar for the performance of a consolidant. It should:

- significantly improve the measurable mechanical properties of the decayed stone, such as compressive strength and abrasion resistance
- slow down the rate of deterioration
- ensure that treated stone has similar thermal and moisture expansion characteristics to untreated stone
- allow for future retreatment with the same or a different consolidant

It should not:

- significantly affect the pore structure of the stone, nor moisture transfer within it
- create an interface between treated and untreated areas
- form harmful by-products
- significantly alter the appearance (colour, texture or surface reflectance) of the stone
- promote or support microbiological growth
- prevent other treatments, such as cleaning, repointing or mortar repair

Working properties

A good consolidant should have low viscosity to allow deep penetration and uniform deposition within the stone. It should solidify within a reasonably short time. Deposition should occur at depth and the consolidant should not be drawn back towards the surface as the solvent or carrier evaporates. Ideally, a consolidant should also have low toxicity.

The effectiveness of a consolidant always depends on the properties of the stone being treated, including its pore shape and connectivity, pore-size distribution, and its chemical makeup.

Modern consolidants

Since the 1970s, penetrating stone consolidants used in the UK have been one of two types: water based (for example limewater and aqueous dispersions of silica) and organic-solvent based (for example silanes and acrylic resins).

In the 1970s and 80s, some conservators favoured the use of limewater. This is made by vigorously mixing lime putty with water. When allowed to rest, most of the lime crystals settle to the bottom of the container, but some dissolve, forming a colourless saturated solution of calcium hydroxide. This solution can be applied onto the decayed stone, where it is absorbed into the friable material. Subsequently, the dissolved lime reacts with the carbon dioxide in the air to form calcium carbonate.

Limewater has one major drawback: calcium hydroxide has limited solubility, with a maximum concentration of about 1.6g of lime in 1 litre of water. Thus, for effective consolidation large quantities of limewater are often needed; this may initiate harmful side effects, such as dissolution, mobilisation and re-crystallisation of salts.

Many conservators and specifiers had little confidence in the efficacy of limewater and so looked to other consolidants. In the 1970s, there was an increased awareness of the potential for chemical consolidants, in particular, alkoxysilanes. These have low viscosity and good penetration, and form stable and strong silica bonds within the pores of the stone. The most widely used alkoxysilanes were based on methyltrimethoxysilane (MTMOS). However, these consolidants also had drawbacks. It took days, or even weeks, for alkoxysilanes to solidify. This long period of time allowed evaporation to draw much of the consolidant back towards the surface, so there was very little consolidation at depth.

To overcome this, in 1975 the Building Research Establishment (BRE) developed Brethane.
This was a modified MTMOS, containing a lead catalyst, which enabled it to set within a few hours. Trials (reported in 2002), however, showed mixed results. While Brethane worked in some situations, in others decay continued and stone became discoloured. In any case, due to concerns over toxicity because of its lead content, Brethane fell out of use in the 1980s. Other alkoxysilanes remained in use. However there is a problem in trying to consolidate calcareous stone with material based on silica. Because of differences in their chemistry, there is a poor bond between silica and the host stone. Thus, the silica gel that is deposited may fill the pores of the stone but does not necessarily improve cohesion.

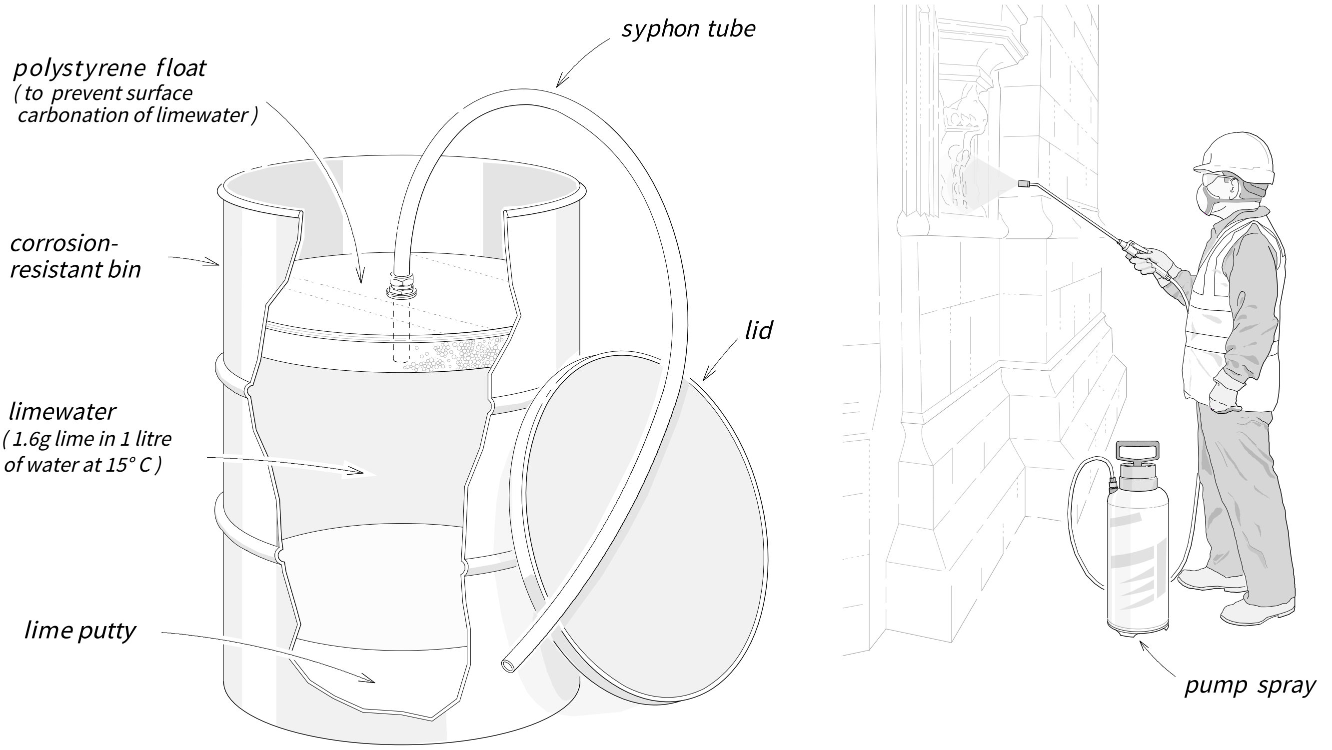
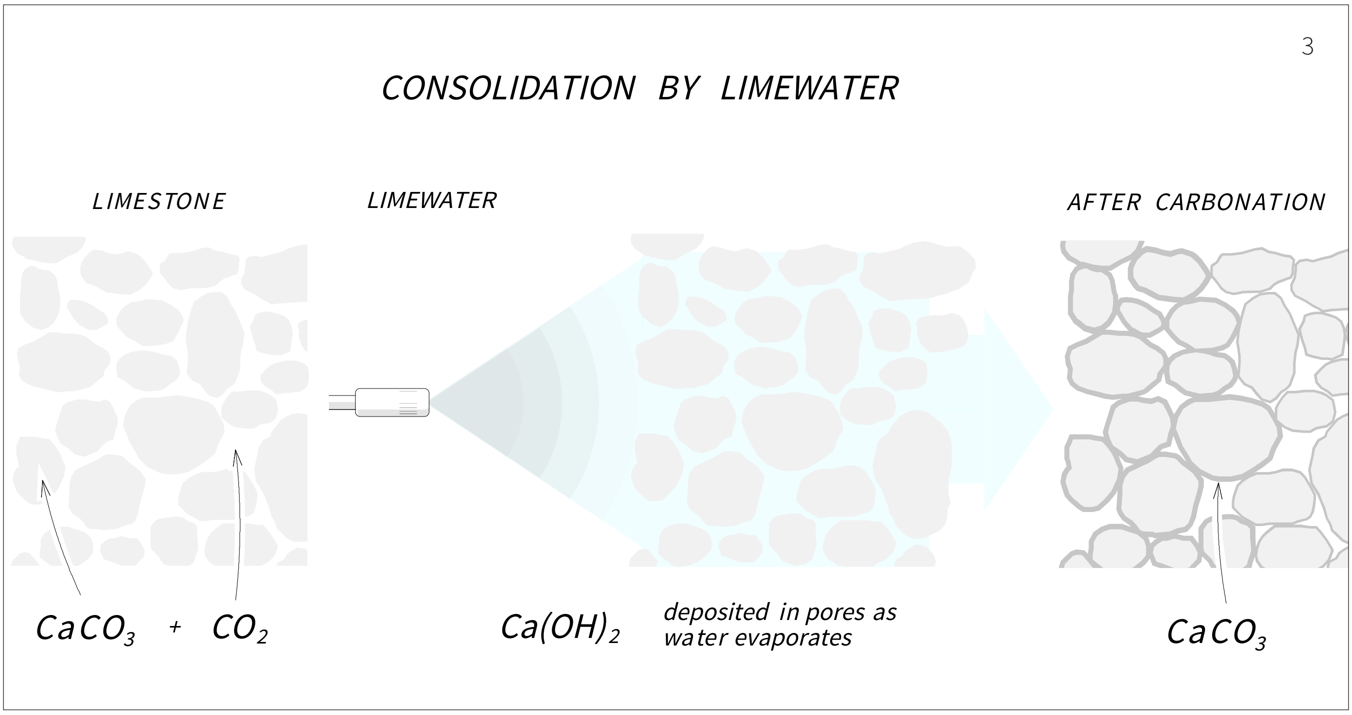


Figure 2
Diagram showing the equipment and method for limewater storage (left) and application (right).



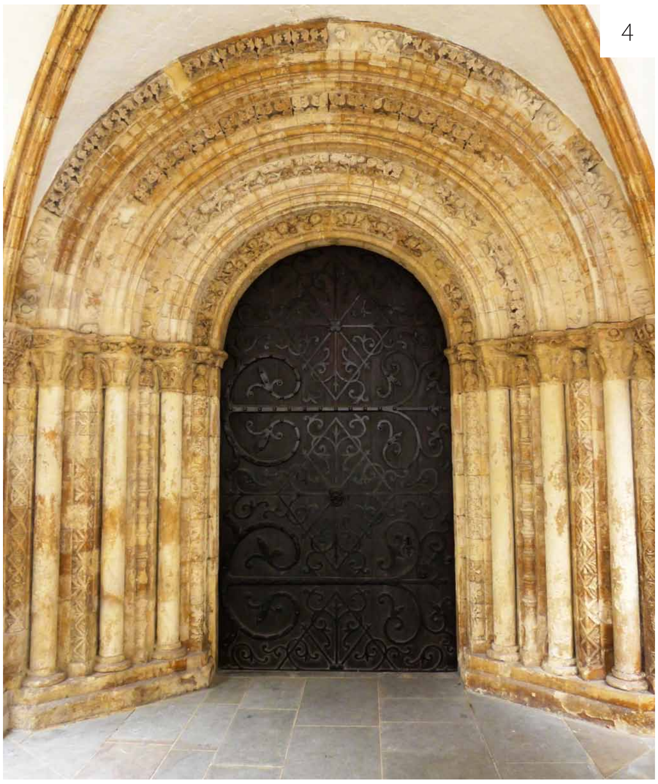


Figure 3
Diagram showing the theoretical principles of limewater in which calcium hydroxide in solution is delivered into the calcareous stone where it carbonates by reacting with carbon dioxide in the presence of water.

Figure 4
West Door of Temple Church, London. This had Romanesque origins and was substantially restored in the 1840s. Since then, the decay of Caen stone has continued. As a result, it has been subjected to a number of treatments with consolidants including 'Hemingways Patent Siasic process' (a mixture of potassium silicate and arsenic acid) in 1913, silicon ester in 1927, and limewater to some areas and Brethane to others in 1984.

In the past 20 years, there have been many developments in stone consolidation. These include the use of fluoropolymers and the modification of existing consolidants (such as MTMOS) to improve performance. Inorganic pre-treatment based on tartaric acid (known as Hydroxylating Conversion Treatment [HCT]) has also been used to try to improve the bond between calcite materials and silanes. Despite these developments, there has been a reluctance to use organic-solvent-based systems on external weathered stonework in the UK, as not much is known about their long-term effect.

Development of nanolime

Nanolime was originally developed in the 1960s for the surface consolidation of frescoes. In the early 2000s, it was seized on as a stone consolidant, and began to be widely used. However, although there had been some laboratory-based research about nanolime (notably the Stonecore project) and there was some experience of its use on internal stone in controlled environments, there was little evidence relating to its use and effectiveness in external situations, particularly in the UK. Furthermore, there was very little guidance on when and how it should be used, and specifications varied widely. Also, in some early examples of its use, it was prone to forming a white bloom on the stone. This raised concerns that nanolime did not penetrate deeply, but instead was concentrated on or close to the surface.

As a result, the Building Conservation and Research Team of Historic England (then English Heritage) commissioned the University of Bath to carry out laboratory and site-based investigations in order to:

- understand how nanolime acts as a consolidant for decayed limestone
- observe its effect on site-based trials
- understand the parameters for optimising its performance
- develop guidelines for site use

The information in this advice note is based on the results of this research. It also draws on the experience of a number of conservators who have used nanolime, and on discussions held during a symposium held at the University of Bath in September 2015.

1 Nanolime

1.1 What is nanolime?

Nanolime is a dispersion of synthesised calcium hydroxide in an alcoholic medium. Nanoparticles are defined as being particles of any shape, less than 100 nanometres (0.1µm) in any one dimension. A typical dispersion of nanolime contains plate-like crystals of calcium hydroxide that are less than 100 nanometres thick, but may be up to 400 nanometres wide.

Nano-sized crystals can also occur in lime putty made by slaking quicklime; during storage of lime putty, crystals of calcium hydroxide repeatedly dissolve and re-crystallise into smaller crystals, so that after a long period (perhaps 20 years) a substantial proportion of them are nano-sized. This accounts for the fact that well-aged lime putty is more reactive and more plastic than fresh lime putty.

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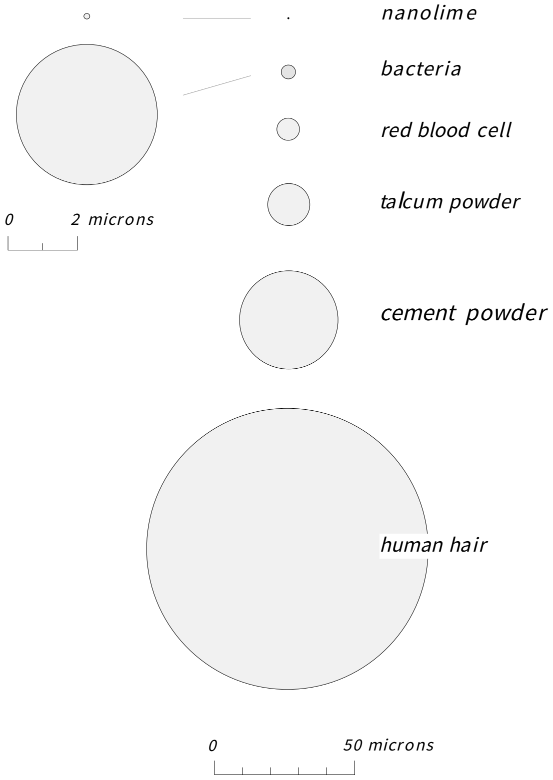

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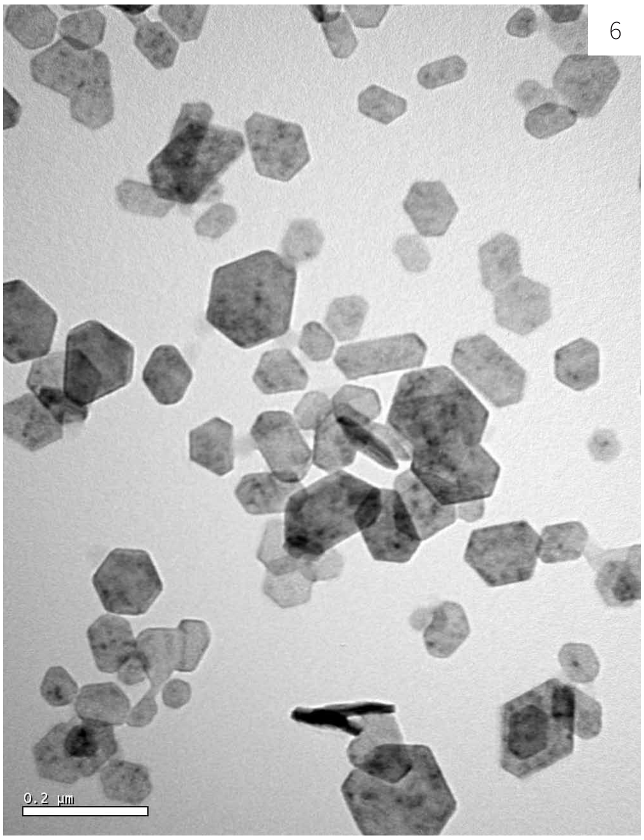

Figure 5
Diagram showing the size of a particle of nanolime compared to other materials.

Figure 6
Transmission Electron Microscope (TEM) image of nanolime crystals showing the hexagonal plate shape.

An important step in the development of nanolime was the work carried out by Giorgi, Dei and Baglioni (reported in 2000). They used calcium hydroxide as a dispersion of natural lime putty in isopropanol rather than as a solution in water (as in limewater). By doing so, concentrations of calcium hydroxide up to three times higher than in limewater could be delivered onto the substrate.

The second development (reported in 2001) was to use a synthetic form of calcium hydroxide. The conditions for synthesis of this material can be controlled to grow nano-sized crystals. As part of the process, water is substituted with alcohol so that the resultant calcium hydroxide crystals are dispersed in the alcohol. This is not an easy process, as there is a tendency for the crystals to agglomerate. The amount of calcium hydroxide in the dispersion can be varied, so different concentrations from 5 g per litre up to 50g per litre are available.

There are a number of benefits of using alcohol as the dispersion medium. Alcohol wets the stone effectively, allowing for deep penetration. It improves particle stability and readily evaporates, ensuring rapid deposition of the calcium hydroxide. Furthermore, it does not act as a solvent for salts or minerals within the stone, and is less toxic than other organic solvents.

The type of alcohol used for lime dispersions can be varied; ethanol, n-propanol and isopropanol have all been used. Different alcohols have different evaporation rates due to the size and shape of their molecules (see Table 1).

Evaporation rates (compared to evaporation rate of butanol)	
Ethanol	1.4
N-propanol	1.3
Isopropanol	2.83

Table 1

Evaporation rates of alcohols. These are comparative rather than absolute values and are expressed against a standard of butanol, which has an evaporation rate of 1. Higher values mean faster evaporation.

Figure 7

Bottles of nanolime.

Nanolime consolidants are generally labelled according to the type of solvent and the concentration of calcium hydroxide; for example, the product label E5 represents 5g per litre of calcium hydroxide in ethanol and IP25 is 25g per litre of calcium hydroxide in isopropanol.

The two product ranges most commonly used in the UK are:

CaLoSil manufactured by IBZ Salzchemie GmbH, Germany (http://www.ibz-freiberg.de/en/). The products are available in the UK through Hirst Conservation (www.hirst-conservation.com/). This is currently available as E5, E25 and E50; IP5, IP15 and IP25; NP5, NP15 and NP50.

Nanorestore Plus® manufactured and distributed by CSGI, Italy (http://www.csgi.unifi.it/products/plus.html). This is currently available as E5, E10, IP5 and IP10.

1.2 How does nanolime work?

Theoretical performance

One litre of nanolime E25 contains some 3000 million nano-sized calcium hydroxide crystals. For optimum consolidation, the maximum number of nanolime particles must be drawn into the stone and penetrate the pore network. As the alcohol evaporates, the calcium hydroxide must then be deposited at sufficient depth for consolidation to take place through the whole zone of deterioration and into the sound stone behind. Under optimum conditions, the nanolime will then carbonate in the presence of moisture, as shown in the simplified carbonation reaction below:

$$\mathrm{Ca(OH)_2 + CO_2 \longrightarrow CaCO_3 + H_2O}$$

Conversion into calcium carbonate relies on a thin film of water forming on the surface of the calcium hydroxide crystals. This must be sufficient to facilitate the dissolution of carbon dioxide from the air to form carbonic acid. To a large extent, this depends on the characteristics of the host material and the environmental conditions.

Initially, calcium carbonate forms a thin coating on the existing grains of stone, but further carbonation leads to partial filling of the pores with calcium carbonate. Effective consolidation is dependent on these crystals of new calcium carbonate bonding to both the host stone and to one another to form a strong matrix.

Limitations on performance

The three parameters considered most relevant in determining whether nanolime is effective as a consolidant are:

- the degree of penetration and deposition
- the degree of carbonation
- the cohesion of the new calcite matrix and its bond with the host stone

For effective consolidation, both the alcohol and the calcium hydroxide in nanolime should penetrate to the required depth. However, laboratory tests have shown that this is not generally the case; although there might be considerable penetration of the alcohol, there is not the same degree of penetration of the calcium hydroxide particles.

Furthermore, although presented in the lime cycle as a simple chemical equation, carbonation is a complex process that involves various different mineral phases. In the case of nanolime, recent research has shown that under humid conditions (80 ±5% RH) and at room temperature, carbonation starts with the formation of amorphous calcium carbonate (often referred to as ACC). If alcohol is present, the ACC partly changes into two different metastable forms of calcium carbonate (vaterite and aragonite) that will subsequently (through a process of dissolution and precipitation) re-crystallise as calcite (a more stable form of calcium carbonate). This process starts to occur within minutes of the nanolime being applied, but can take several days or weeks to complete. The complex phase changes in the crystalline structure of calcium carbonate might explain the unexpected reactions of some treated stone: early increase in strength is sometimes followed by a reduction in the next few months, although sometimes that reduction is followed by a subsequent increase.

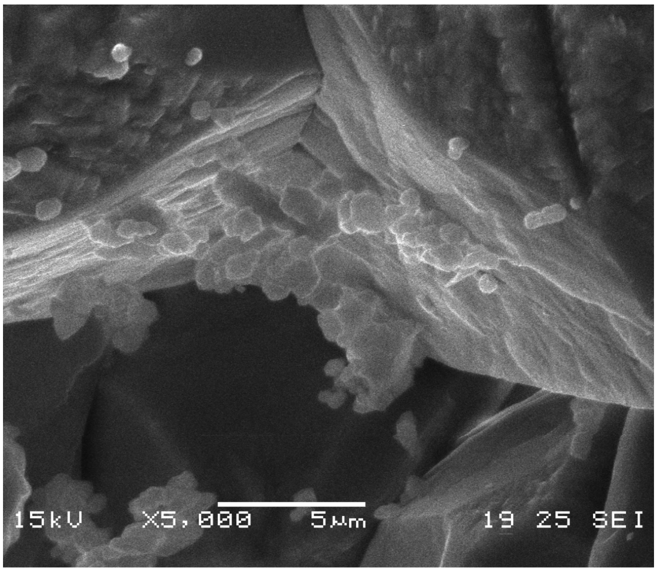

Figure 8
Scanning Electron Microscope (SEM) image showing nanolime particles deposited on the internal pore walls of Bath stone (Magnification x5000).

1.3 Storing nanolime

Nanolime is a colloidal dispersion; that is, a mixture in which the solid settles extremely slowly or not at all. When fresh, nanolime is a thin translucent milky liquid, but if allowed to rest without being shaken, a certain amount of white material will settle at the bottom of the bottle. If the container is shaken, part of the solid material will return to suspension.

Transmission Electron Microscopy (TEM) has shown that over a period of a few weeks, as well as settling, individual particles of calcium hydroxide often clump together. Once this agglomeration occurs, the action of shaking cannot separate the particles, and the benefit of a 'nano' material is lost. Furthermore, after synthesis, changes in the chemical and physical properties of calcium hydroxide can occur, which can affect the performance of nanolime: the sharp hexagonal shape of the particles may be 'softened' and the calcium hydroxide may react with the alcohol to form calcium alkoxides. The presence of these alkoxides seems to change and delay the carbonation process.

Nanolime is therefore most effective when fresh. Bottles should be kept firmly closed to prevent contact between calcium hydroxide particles and air. Nanolime should not be applied after the 'use by' date.

1.4 Diluting nanolime

Nanolime can be diluted to lower its concentration. For example, E25 can be diluted to make E5 by adding one part of E25 to 4 parts of ethanol. Alcohols are available in various forms, such as analytical, laboratory and general use. They all have different degrees of purity, but for diluting nanolime, the anhydrous laboratory grade should be used, as it contains least dissolved water.

Figure 9
Inverted bottle of nanolime showing how, after storage, particles settle at the bottom of the bottle. If allowed to accumulate for too long, particles will agglomerate and shaking will not fully separate them.

2 Suitability of Stone for Treatment

2.1 Stone mineralogy

Due to the chemistry underpinning the use of nanolime, it is thought to be most suitable for consolidating calcareous materials such as lime mortar and limestone. Testing carried out at the University of Bath as part of the Historic England research programme also indicated that calcareous sandstone (sandstone in which the predominant cementing material is calcite) is potentially suitable for consolidation with nanolime. However no testing has been carried out on other sandstones, and there is no evidence that nanolime is effective on such stones. Previous experience suggests that there is likely to be poor bonding between the silicate stone particles and the calcitic consolidant, somewhat analogous to that of silane consolidants applied to limestone.

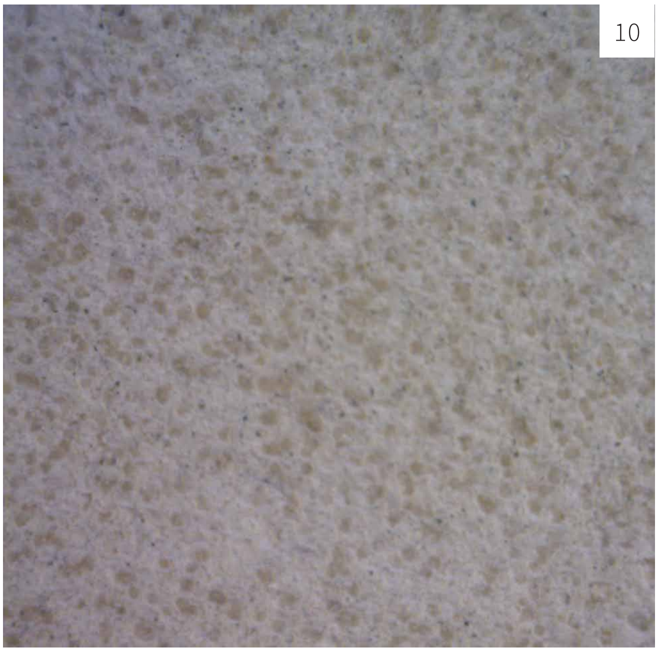
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Figure 10
Magnified view of a microporous stone (clunch) in which the pores are extremely small (invisible at this magnification) but well connected. Magnification: x50.

Figure 11
Magnified view of a porous stone (Ketton) with obvious large pores. Magnification: x50.

2.2 Pore structure of stone

Nanolime is a dispersion, not a solution, so consolidation relies on crystals of calcium hydroxide penetrating to a considerable depth through the pores of the stone. The effectiveness of this penetration depends, among other things, on the size of the crystals and the pore structure of the stone being treated. There are two elements of pore structure that are particularly important; the proportion of the stone that consists of pores (porosity), and the degree to which the pores are connected and therefore able to permit movement of liquid through the stone (permeability).

Both stone and mortars typically have an average pore diameter of 0.1μm–10μm, but the pores in stone vary more greatly in terms of size. Some pores are even smaller than a nano-particle (<0.1μm). The pore-size range for mortars depends entirely on the constituents, but they tend to have much greater connectivity than limestone.

2.3 Type of stone decay

Many types of decay affect external weathered stone surfaces. These include fracturing, cracking and splitting through to erosion, spalling, delamination and exfoliation. The types of decay tend to be peculiar to each type of stone. An illustrated guide to the various types is included in the Historic England *Practical Building Conservation* volume on Stone.

Treatment with nanolime is not appropriate for every kind of decayed stone. Stone suffering from decay that involves separation between adjacent pieces of stone (for example, delamination or cracking) is not suitable for consolidation with nanolime marketed as a consolidant (or indeed any other consolidant) as the number and size of the calcium hydroxide crystals are insufficient to bridge large gaps. (There are related nanolime-based products on the market designed for gap filling, but their use is beyond the scope of this advice note.)

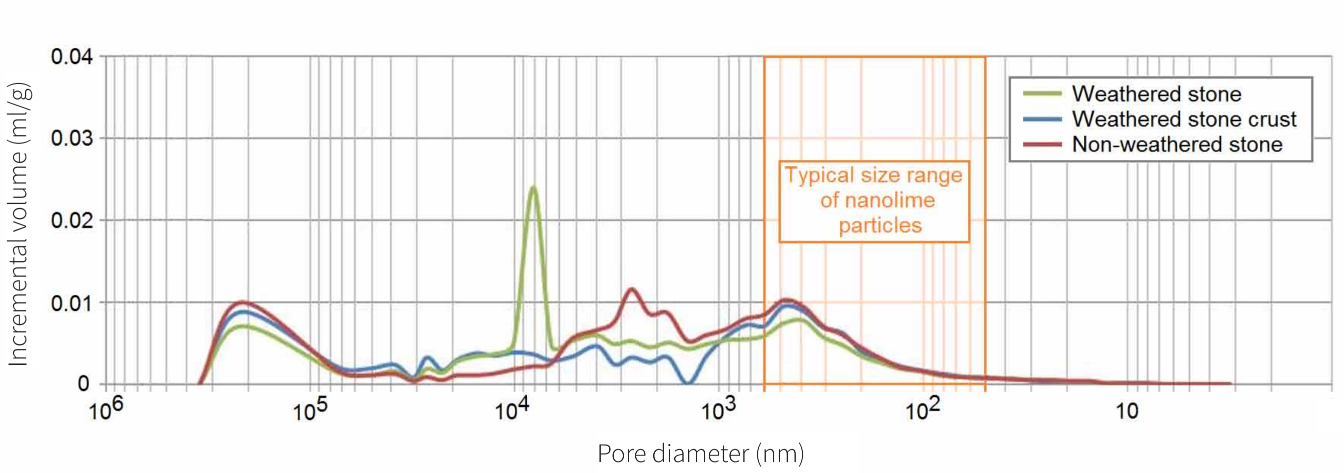

Figure 12
Mercury intrusion porosimetry graph showing the typical pore-size distribution of Bath stone (both weathered and unweathered) and the typical size range of nanolime particles. This shows that some of the particles of nanolime are larger than some of the pores, and therefore will be unable to penetrate into the stone.

Nanolime may be effective for consolidating stone that is crumbling, flaking, or suffering granular disintegration due to dissolution of the calcite binder. These types of decay can affect just the surface (perhaps to a depth of a few mm) or a much greater depth of the stone (typically up to 10–15mm). For consolidation to be effective and durable, the consolidant must penetrate beyond this zone and bond to the sound stone behind. This will ensure that there are no interfaces between decayed and consolidated material.

2.4 Surface condition

Microbiological growth

The surface of weathered stone is often colonised by microbiological growth, usually in the form of algae or lichens. Although many of these are not harmful, some can cause degradation and disaggregation of stone. They can become established on both sound and decayed surfaces, affecting the surface permeability and moisture content of the stone; this in turn will affect the penetration and hence the effectiveness of nanolime treatment.

Surface coatings

Paint and other applied surface coatings (for example, historical treatments such as linseed oil or waterglass) are also likely to affect the application and penetration of nanolime. If these coatings are impervious, they may encourage deterioration of the stone substrate. In some cases, it may be desirable or necessary to remove such coatings to allow consolidation of the exposed stone. If the coating cannot or must not be removed, nanolime should not be applied, as it will not be able to penetrate the coating into the decayed stone.

Surface crusts

Many limestones in England develop a surface crust. This can occur when minerals within the stone migrate to the surface, or calcium carbonate reacts with pollutants such as sulphur dioxide to form calcium sulphate. The latter is often referred to as a gypsum crust and is generally black due to encapsulation of airborne particulates.


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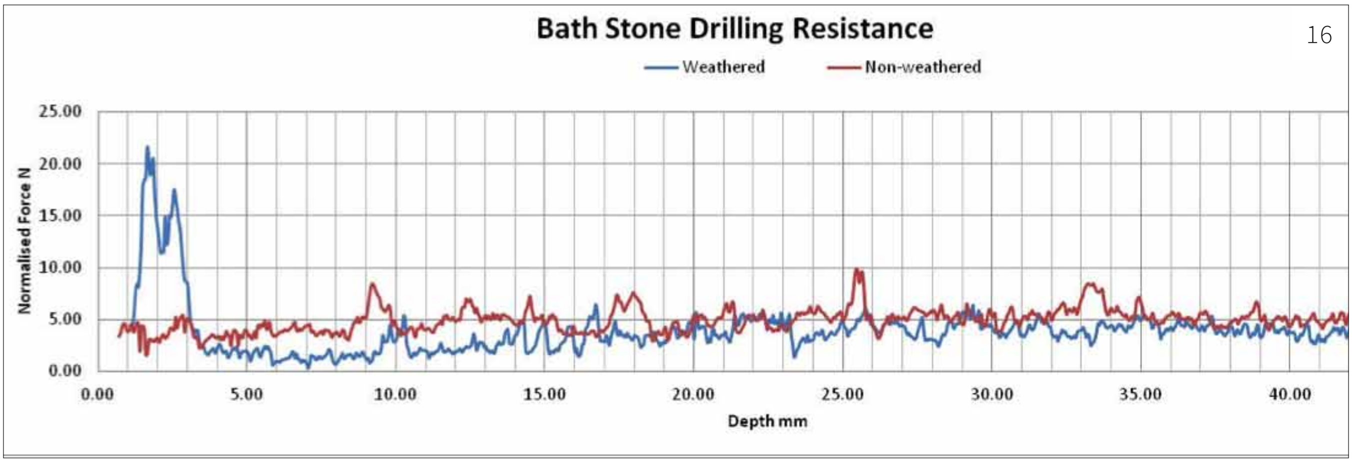
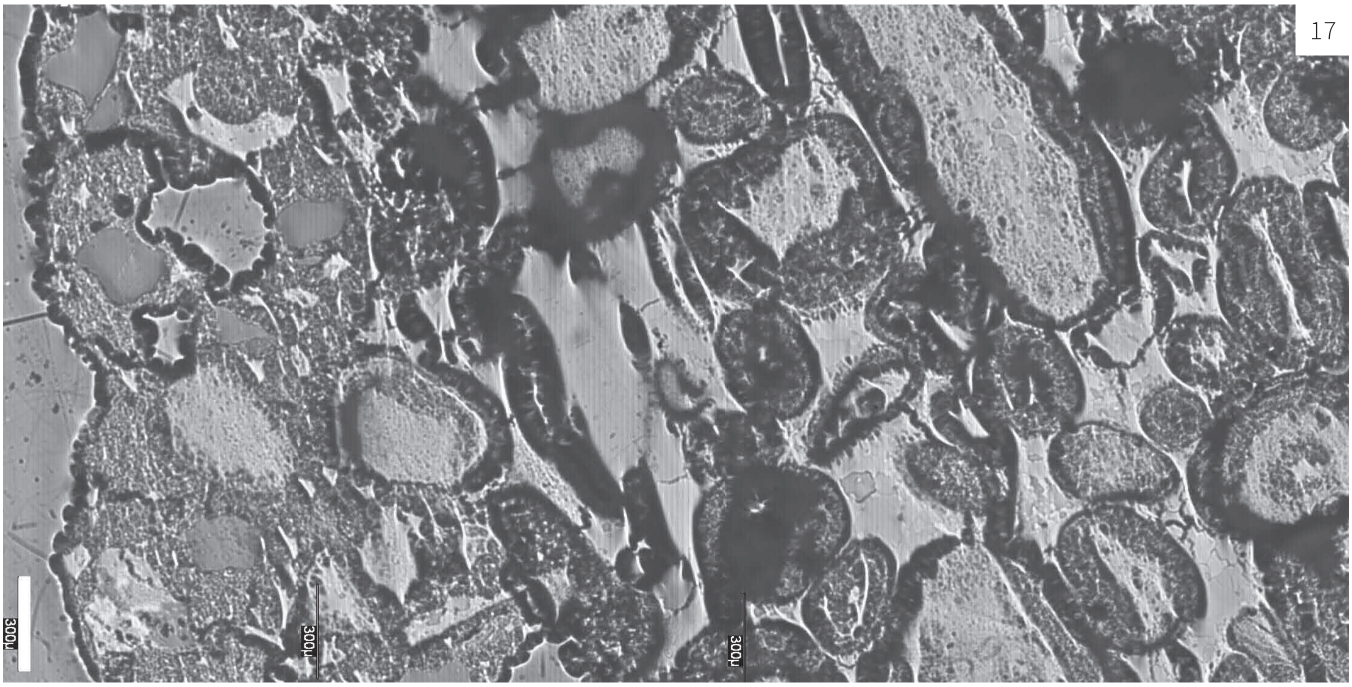
Figure 13
Tewkesbury Abbey, Gloucestershire. Disaggregation of stone behind a hard crust. Although the powdering stone may be suitable for consolidation with nanolime, the gap between the decayed stone and the crust will need to be filled with other material.

Figure 14
Canterbury Cathedral, Kent. Typical decay of Caen stone with spalling surface crust, freshly decayed stone and microbiological growth.

Figure 15
Kensal Green Cemetery, London. Typical section of limestone showing black gypsum crust in the area unwashed by rain, decayed corner with powdering stone and front face which also may have a crust but is washed clean by rainwater.





Gypsum crusts are more or less impermeable. Various mechanisms of decay (such as salt crystallisation) take place at the interface between the crust and the more permeable stone beneath. This can result in binder depletion and powdering of the stone behind the crust. To consolidate this friable material layer, the nanolime must penetrate the surface crust. Tests show that this is very difficult, if not impossible, to achieve. In fact, in most cases, application of nanolime further strengthened the crust creating an even more marked contrast between the surface and the stone beneath.

Figure 16
Drilling resistance of weathered and unweathered Bath stone, showing a large peak in the weathered stone up to a depth of about 3mm caused by the formation of the hard crust.

Figure 17
Cross-section through polished sample of Bath stone showing the hard sulphated crust at the surface, a weakened (darker) layer behind and the less-affected stone on the right. Magnification: x 50.

2.5 Moisture content

The moisture content of the stone is also an important factor in the efficacy of nanolime. Although some moisture is required for carbonation, too much moisture means that pores are either lined or filled with water molecules, which may limit the penetration of nanolime and its deposition in the required location.

Furthermore, where there is moisture, there are also likely to be salts. Salt crystallisation is a primary cause of stone decay, so stone subject to repeated cycles of salt crystallisation may require consolidation. Although the alcohol used as the medium in nanolime will not cause dissolution or recrystallisation of these salts, their presence within the pores of the stone may reduce the ability of nanolime to penetrate or carbonate on the pore wall. Nanolime may therefore be less effective in consolidating stone contaminated with salts.

2.6 Assessing the suitability of stone for treatment with nanolime

In the light of the criteria cited above, when assessing whether a stone is suitable for treatment with nanolime, it is important to consider the following:

- Is the decay of the stone suitable for this treatment (powdering, granular disaggregation rather than cracking/ delamination)?
- Is there an applied surface coating or biological growth that will reduce the ability of nanolime to penetrate the stone? If so, can it be removed (see below)?
- Is powdering or friable stone exposed, or is it covered by a surface crust?
- Is surface absorption sufficient to allow nanolime to penetrate? (This may require an assessment of both the sizes of the pores and their connectivity)

It is unlikely that these conditions will occur over large expanses of stonework, but are more likely to apply to localised areas.

Assessing surface absorption

It is difficult to assess the pore structure of stone on site. There is a complex correlation between porosity and permeability, and any calculation requires information on grain size, surface area and other parameters that are unlikely to be measurable on site. However, some information may be gained just by performing a simple surface absorption test. The most basic method is to carry out a water droplet test in which a droplet of water is placed on the stone surface with a pipette or syringe. If the droplet remains on the surface for a substantial period (perhaps 30 seconds or more), then either the stone is sound or there is a surface crust or some other deposit reducing surface absorption, which would also limit penetration of nanolime. If the water droplet disappears quickly, nanolime may be an appropriate treatment, assuming other criteria are fulfilled. The degree of absorption can also be indicated by the area of wet stone after absorption has occurred; a less absorbent surface results in a larger area of wet stone. A more accurate assessment of surface permeability can be carried out using a Karsten tube.

Figure 18
A simple test for surface absorption involves applying a single droplet of water on the surface using a pipette. The stone on the left is decayed and more porous so the water is quickly absorbed and the wetted area is small; the stone on the right has a surface crust and is less absorbent so the droplet has spread and wets a larger area of the surface.

Figure 19
Karsten tube being used to measure surface permeability. The mouth of the tube is sealed against the stone using plasticine. The column is filled with water level with the top mark of the scale. The level of the water is then noted after specific periods of time, enabling the volume and speed of water absorbed into the stone to be measured.

Figure 20
Sliding a sheet of white paper behind the tube makes it easier to read the level of the water against the scale.

Figure 21
Results from a Karsten tube test can be plotted on a graph to show the amount of water absorbed against time; very permeable stones have a steep curve.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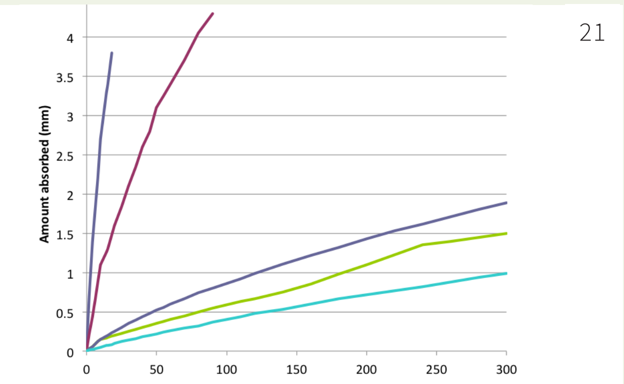
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3 Applying Nanolime

3.1 Extent of application

Research carried out by Historic England and the results from experienced conservators suggest that nanolime should be thought of as a targeted, localised treatment for decayed stone. It should not be used as a general treatment for large areas of stonework.

3.2 Preparation of the substrate

When treating stone with nanolime, it may be necessary to carry out pre-treatment to ensure optimum results. This can involve one or more of the following operations.

Defrassing/rationalisation

Surface decay can lead to both powdering and spalling (separation of small fragments) of the stone surface. Since nanolime consolidants cannot bridge gaps between adjacent pieces of stone, spalls will generally have to be removed prior to applying nanolime to the powdering stone. The process of removing spalls is generally referred to as defrassing or rationalisation. It involves rubbing the surface with a gloved hand or brushing with a stiff bristle brush. Defrassing can also remove powdering stone (i.e. material that might benefit from treatment with nanolime), so a decision to defrass must only be made after careful assessment of the condition of the stone, as well as with due regard to its historic significance or decorative detail. Defrassing should not be carried out as a matter of course and is seldom appropriate for carved stone.

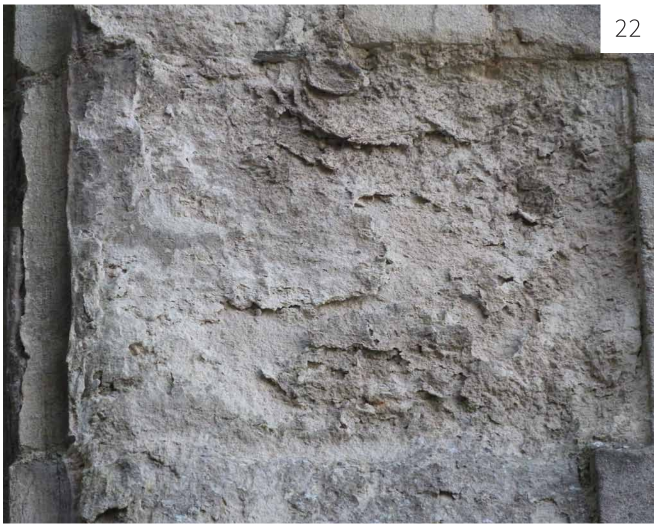
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Figure 22
Stone before de-frassing or 'rationalisation'.

Figure 23
After defrassing to remove surface spalls that are detached from the host stone and so may not be suitable for consolidation. This approach should not generally be taken with decorative or carved stone.

Surface cleaning

This is not normally required prior to treatment with nanolime as decaying surfaces that require consolidation are not usually sufficiently stable to become soiled. Sound surfaces tend to accumulate dirt, but these do not normally require consolidation. However, if the stone has developed a surface crust, the material beneath may be very friable and in need of consolidation. In such cases, softening or reducing the surface crust may aid penetration of nanolime; this is usually best achieved by the application of a poultice containing a suitable chemical such as ammonium carbonate.

Removing biological growth

Microbiological growth is often found on both sound and decayed stone. Although algae grow on the surface, lichens can have fungal roots (hyphae) that penetrate deep into the stone. The most effective way to remove microbiological growth is to change the environment of the stone. This can be accomplished by either drying it out, excluding natural light, removing nutrients, or exposing it to UV light. Once the growth has died, it can be brushed off. Unfortunately, these methods can take several weeks, so removal usually involves brushing, steam cleaning and in some cases, the use of biocide.

3.3 Methods of applying nanolime

There are three main ways in which nanolime is applied to external limestone: spray, brush, and pipette or syringe. The most appropriate may well depend on the personal preference of the conservator. A fourth method (total immersion) is not considered practical or viable for the treatment of external weathered stone.

The coverage of nanolime depends on the type of stone and its condition. Carefully measured application of E25 on weathered Bath stone showed that the stone absorbed approximately 2.5 litres per m^2 for the first application, and lesser amounts on subsequent ones.

Health and safety

Nanolime should always be handled with care, following the same procedures that would apply to the use of ethanol or other alcohols. It must be used only in well-ventilated spaces and with the appropriate personal protective equipment (goggles and gloves). Nanolime is flammable, and must not be used in the presence of flames and sparks.


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Figure 24
The use of a syringe allows an accurate record to be kept of the amount of nanolime applied.

Figure 25
Nanolime being applied by syringe with a sponge held beneath to catch any excess.

Spray application

This involves using a standard hand-held sprayer to spray nanolime onto the target area. A clean, damp sponge should be held under the area being treated to soak up any runoff. A potential disadvantage of this method is that the spray zone may be too wide for precise targeted application. There is also a risk that there will be some evaporation of the solvent before the nanolime reaches the stone surface. This increases the likelihood that it will be deposited on the surface rather than penetrate the stone.

Application by brush

A small brush or water brush (pipette with integral brush attachment) can be used to apply nanolime in a controlled manner. However, the physical contact between brush and stone may disrupt the deteriorated surface. As is the case with spray application, a clean, damp sponge should be used to soak up the runoff.

Application by pipette or syringe

Many conservators favour this method, as it can be precisely targeted but does not disrupt the stone surface. The nanolime is gently dribbled onto the decayed stone surface until no more can be absorbed. Again, a sponge should be used to soak up the excess. For large areas, the syringe or pipette is moved from side to side over the surface, starting from the top; maintaining even pressure on the plunger or bulb ensures a steady flow of nanolime.

Figure 26
Norwich Cathedral. Conservator using a water brush to apply nanolime to localised decay of carved detail in the cloisters.

3.4 Number and timing of applications

Nanolime is initially readily absorbed. Application should continue until the surface glistens and no more nanolime can be absorbed. This normally takes only a few minutes and constitutes a single application. Subsequent applications should be made only after the nanolime has been completely absorbed, but before the surface has dried.

Although the characteristics of the stone, the degree of deterioration, and the environmental conditions will all dictate the amount of nanolime that is necessary, most stones should receive no more than three applications. Although the alcohol may continue to be absorbed, stone cannot endlessly absorb calcium hydroxide particles. The carbonation process starts the moment calcium hydroxide is exposed to air within the pores of the stone. Once carbonation becomes established, calcium carbonate crystals start to grow within the pores of the stone, making the pores smaller and further absorption of nanolime difficult, if not impossible. At this stage, any additional nanolime will tend to accumulate on the surface forming a white bloom.

In recent practice, there have been considerable variations in the timing of multiple applications amongst different practitioners. Studies carried out at the University of Bath and at various site trials indicated that for maximum benefit all the applications should preferably be made during one day and definitely over no more than two consecutive days. However, there have been some situations where conservators have found that nanolime continued to be absorbed over a much longer period. It is always important to record the application periods and the quantity and type of consolidant used.

Figure 27
The application of nanolime can lead to the 'blooming' of the surface – usually an indication that too much nanolime has been applied, or the product was too concentrated. Various methods are available to mitigate this effect.

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3.5 Avoiding surface blooming

Sometimes treatment with nanolime causes a white bloom to appear on the surface of the stone. Not only is a white bloom unsightly, but it can result in formation of a hard, impermeable crust that adversely affects moisture transfer and may inhibit adhesion of repair mortars and shelter coats.

Each drop of E25 nanolime contains around 100,000 particles of calcium hydroxide, and as the alcohol is absorbed into the stone it carries a multitude of particles into each pore. Inevitably, this leads to a concentration of particles and clogging of the mouth of the pore. Once the alcohol evaporates, these particles are left on the surface, where they carbonate, forming a layer of calcium carbonate: the white bloom. This is more likely to occur if the more concentrated nanolime products are used. The risk of blooming is also increased if nanolime is applied in warm, dry, windy conditions, or if a large proportion of the pores in the stone are smaller than the nanolime particles. A white bloom may also form if further applications are made on a treated surface whose pores are already filled with deposited nanolime.

Possible methods for reducing surface bloom include sponging the surface immediately after treatment with a clean damp sponge (thus removing excess nanolime) and covering the treated surface with cling film to reduce the rate of evaporation. Whilst these steps may reduce the risk of a white bloom, it should be remembered that formation of a bloom is usually symptomatic of poor penetration or too many applications. Action to improve penetration (see below) is preferable to simply treating the symptom or disguising the white bloom with shelter coat.

Figure 28
Chilmark stone capital on the Chapter House at Salisbury Cathedral used as part of the Historic England trial.

Figures 29, 30
After treatment with nanolime, there was initially an intense white bloom, indicative of excess nanolime accumulating on the surface (Figure 29). Although this was concealed to some extent by shelter coating (Figure 30), it would have been better to have avoided formation of a white bloom in the first place. The conservator reported that “Where Nanolime (E25) was used to consolidate friable Chilmark surfaces, a distinct hardening of these surfaces was noted. The advantage of such surface hardening is that less stone (if any) is required to be removed in order to carry out a successful mortar repair”.

3.6 Encouraging penetration and carbonation

The main challenge in treating a decayed surface with nanolime is to obtain maximum penetration, even deposition at depth, and to ensure complete carbonation. It is impossible to set rules to cover every situation, as there are many variables. There are, however, ways to optimise the effect of the treatment. These methods are not necessarily based on documented research, or even on theoretical principle, but have been found by experienced conservators to work in the field.

Pre-wetting with water

If the stone is dry, then some pre-wetting before applying nanolime will introduce moisture into the pores of the stone. This should aid carbonation. The alcoholic media used for nanolime are all completely miscible with water, and it is unlikely that the presence of limited moisture in the stone will affect penetration of the solvent. However, if the stone is saturated, nanolime will be unable to penetrate the pores and attach to the stone matrix.

Pre-wetting with alcohol

Some conservators report that pre-wetting with alcohol enables nanolime to penetrate stone more easily; however, laboratory tests provided no conclusive evidence of this.

Conservation practice has also shown that flushing out voids with a mixture of water and alcohol prior to grouting is an effective way of encouraging wetting of the substrate. Since tests have not yet been carried out, the effectiveness of this pre-wetting procedure in the use of nanolime is unknown.

Concentration of nanolime

When nanolime was first developed, it was used in low concentrations (5g per litre), as it was feared that higher concentrations would reduce penetration. For reasons that are not clear, E25 (25g per litre) has become the standard product for stone consolidation in the UK. This has proved problematic, as it is now understood that higher concentrations of nanolime can cause surface blooming. For this reason, one manufacturer makes nanolime at a maximum concentration of 10g per litre. Furthermore, research has indicated that applications of E5 achieve deeper penetration than E25. Conservators also report that an initial application of E5 followed by E25 works well, particularly if the cycle is repeated three times within one day (or two consecutive days at the most).

Due to variability of stone type and decay, it is hard to make definitive recommendations, but the general consensus is that a greater number of applications of less concentrated nanolime produce better results than fewer applications of a more concentrated product.

Figure 31
The application of clingfilm to the surface immediately after application of nanolime can reduce the evaporation of alcohol and aid deeper penetration.

Covering treated area after application

One of the problems of using alcohol as a carrier for nanolime is its rate of evaporation. Although nanolime initially penetrates into the stone, once application stops, the direction of movement will reverse as the solvent evaporates, thus reducing the effective depth of penetration. For this reason, and based on the experience of conservators in the field, it is best to keep the application process as constant as possible and avoid allowing the surface to dry between applications. It has been found that mist-spraying with water and covering the treated area with a thin membrane such as cling film immediately after application reduces both the evaporation rate of nanolime and the potential for white bloom. One manufacturer recommends that the treated surface should be covered with cellulose paper pulp soaked with distilled water, which is then removed after it has dried. These are sensible precautions, especially if it is warm and dry or windy at the time of application.

Environmental conditions

Results have shown that nanolime works best in an external environment with relative humidity between 50–100% RH. Although it is not normally possible (or at least feasible) to control the environmental conditions of external weathered stone, the English climate seems fairly well suited for successful nanolime treatment, as long as steps are taken to protect the treated stone from wind, direct heat and freezing temperatures.

Tests have demonstrated that there is no consistent measurable improvement in consolidation when the surface of stone that has been treated with nanolime is lightly sprayed with water in an attempt to aid curing (as opposed to spraying to prevent drying of the surface between applications).

Optimising the performance of nanolime

- Remove or reduce surface crusts, coatings and biological growth prior to applying nanolime
- Use a brush, pipette or syringe for accurate targeted application.
- Apply to damp, but not wet, stone
- Select the appropriate concentration of nanolime for the type of stone and the number of applications
- Always use nanolime as fresh as possible
- Sponge off any excess nanolime during application
- Apply the second and subsequent applications after the preceding one has been absorbed but before the surface has dried
- For most stone types, apply no more than three applications
- Avoid applying nanolime in very warm or windy conditions
- Lightly spray the treated surface with water and cover the treated surface with cling film to reduce evaporation of the solvent, which might draw the nanolime back to the surface

4 The Effectiveness of Nanolime

4.1 Assessing effectiveness

Site treatments are rarely assessed using quantitative criteria; more often a combination of knowledge, appearance and instinct guides conservators, masons and other conservation professionals when assessing the benefit of any treatment.

Many of the published papers regarding nanolime have used increased compressive strength as a measure of its effectiveness. However, consolidation of decaying stone is required in order to increase its durability and prevent or reduce further decay from weathering, moisture or pollution; increasing its compressive strength may be a result of treatment but measuring this does not necessarily provide an accurate assessment of the desired benefits.

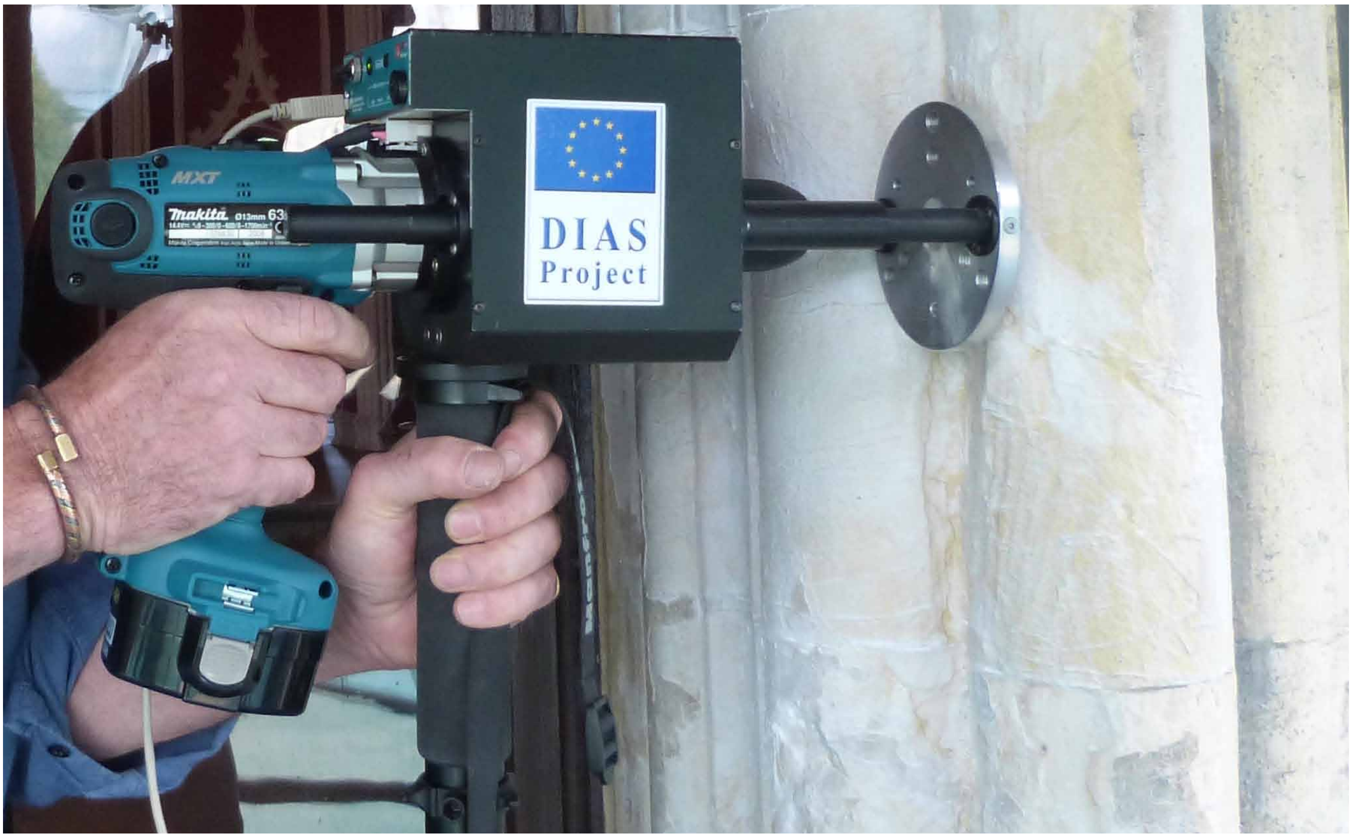
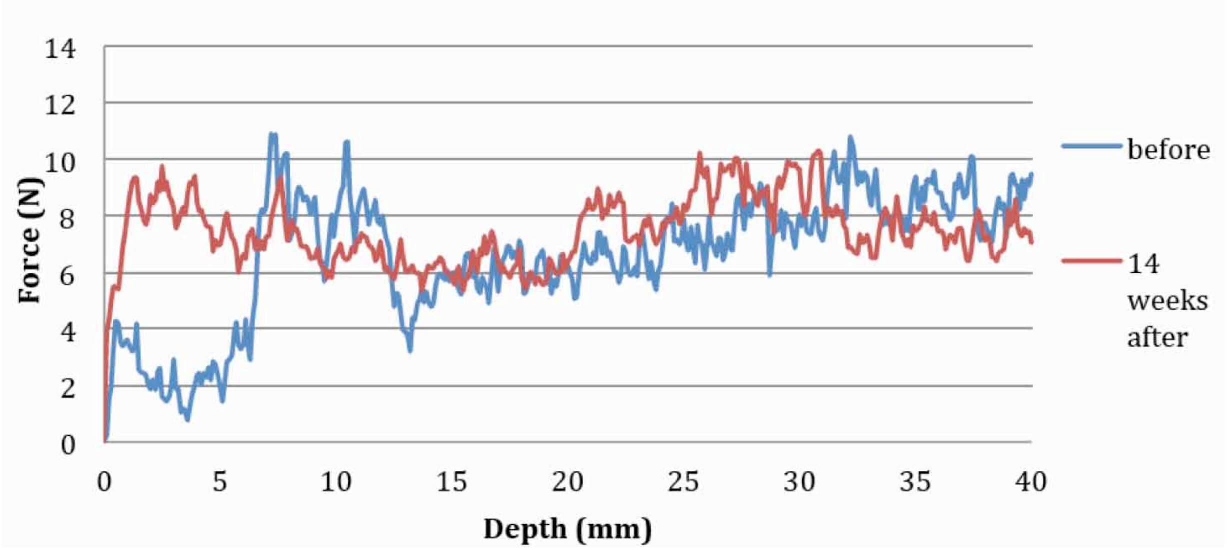


Figure 32
Using the Drilling Resistance Measurement System (DRMS).

Therefore, a more suitable method for measuring the effect of nanolime is to measure drilling resistance, using a system such as the Drilling Resistance Measurement System (DRMS) developed by SINT Technology. This device was used in the Historic England Research project both in laboratory tests and at site-based trials. For all tests, a 5 mm-diameter flat-tipped diamond drill bit was operated at a constant rotational speed of 600 rpm with a penetration rate of 10 mm/min.

The machine measured the force required to maintain these specific values (which is related to the mechanical characteristics of the material tested), and this force is plotted against the depth of penetration. Results before and after treatment can easily be compared and further tests can be carried out subsequently to measure any longer term changes.



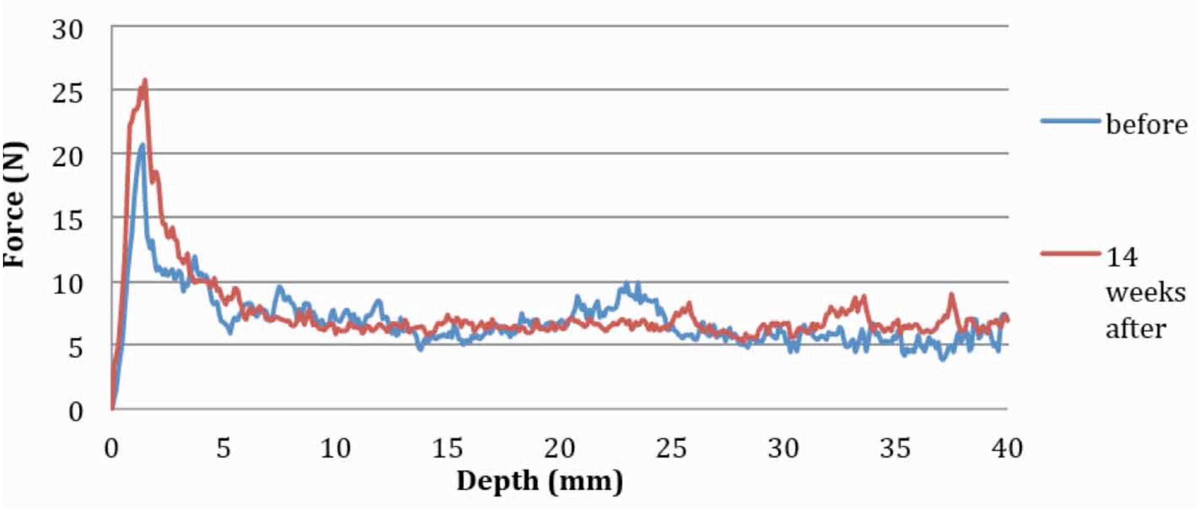


Figure 33
DRM plot showing how the weakened surface of stone can be strengthened (in this case to a depth of 7–8mm) by the application of nanolime.

Figure 34
DRM graph showing how the application of nanolime to a stone with an existing surface crust can cause the crust to become even stronger.

4.2 Research results

The Historic England research project evaluated a number of representative British limestones before and after treatment. Some stones were evaluated soon after treatment while others were evaluated later than that; in some cases, as much as three years after treatment.

Although some of the results were not obviously clear and coherent, they highlighted the important influence of subtle variations in the stone's properties on the interaction with applied nanolime. The conclusions which can be drawn from the research are listed below:

- There is no clear evidence that shaking a bottle of nanolime returns settled and/or agglomerated nanolime particles back into suspension.
- Tests suggest that lower concentrations of nanolime (5 or 10g per litre) penetrate further and result in less surface blooming.
- The amount of nanolime applied does not correlate to the depth of penetration or degree of consolidation.
- Higher humidity promotes carbonation of the nanolime, so application in environmental conditions of RH >65% would be advantageous.
- Nanolime tends to accumulate at the surface on any stone with an existing surface crust that is denser than the bulk of the stone. The result of treatment with nanolime can make the surface crust even stronger and less permeable; this may initiate or exacerbate other mechanisms of decay.
- Nanolime should be applied locally, using a controlled method of application (syringe, pipette or brush) to deteriorated surfaces. It is not appropriate for use as a general surface consolidant.
- Treatment of decayed stone with nanolime can result in a hardened, sometimes glassy, crust; this may affect the adhesion of mortars and shelter coat. For many stones, nanolime appears to effectively consolidate the surface, but penetration is generally restricted to no more than about 5mm. Although this might not extend beyond the zone of decay, it may be sufficient for the requirements of a particular situation; for example, at York Minster, surface consolidation of decayed magnesian limestone was sufficient to allow the application of mortar repairs and shelter coat.
- There is evidence that nanolime applied to salt-laden stones can cause subsurface weakening. It is thought that this occurs because salts tend to crystallise at the interface between treated and untreated stone.
- Limited freeze/thaw tests do not show an appreciable difference between treated and untreated stones.
- Measurements with a Karsten tube suggest that surfaces treated with nanolime show a lower surface absorption compared to untreated stone.
- The depth of penetration depends (among other things) on the capillary forces that draw the solvent into the stone. These compete with evaporation, which moves the solvent to the surface. To reduce evaporation, spraying with water and covering the treated area immediately post treatment can be beneficial, especially in warm or windy conditions.
- There is no advantage in applying nanolime over an extended period of time (such as many days or weeks). Tests and site-based experience suggest best results (and cost-effective application) can be achieved by up to three applications, ideally carried out in one day, but at the most, over two consecutive days, without allowing the surface to dry between applications.

4.3 Long-term effectiveness

Although nanolime has been available for a number of years, its long-term effectiveness as a stone consolidant has not yet been evaluated. Results from research in Europe and elsewhere have shown that in some cases, treated stones continued to develop strength over several months; in others, strength gain levelled off. In some, an initial increase in strength was later lost.

In many situations where it has been used in the UK, nanolime has been part of a process that also involves treatment either with lime mortar or shelter coat or both. This makes it impossible to isolate and quantify the effect of the nanolime. However, there are a couple of programmes of formal testing currently underway, in which the performance of nanolime is being measured against other stone consolidants. Additionally, the stones used in the Historic England research programme have been retained, and will be tested again in the future to evaluate long-term effects.


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Figure 35
York Minster. This area was chosen to carry out trial repairs of the magnesian limestone. The results were sufficiently good to allow conservators to adopt nanolime as a pre-treatment (prior to mortar repair and shelter coat) for decayed areas of stone on the East Front of the Minster.

Figure 36

Trials of various consolidants (including nanolime) have been carried out on the remains of Reigate stone columns at Westminster Cathedral. These are part of the long-term monitoring of the effect of nanolime.


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5 Conclusion

Nanolime has been used as a stone consolidant for over ten years in the UK, with varying degrees of success. It is now apparent that, measured against the criteria detailed at the beginning of this advice note, nanolime has not been the panacea for stone deterioration that many people were expecting (or at least hoping) it to be.

However, for some stones under certain conditions, it is possible for nanolime to consolidate to the required depth. For other stones, the consolidating effect is limited to the surface. In some cases, this may be adequate if it provides sufficient stability for subsequent conservation interventions, especially mortar repair and shelter coat. Nanolime therefore should be considered (along with other consolidants) as a potentially useful part of the conservation tool bag; it certainly has a part to play. It should only be used following consideration of other steps (including preventive measures) that might be appropriate for slowing the rate of deterioration of the stone.

If nanolime is applied in accordance with the guidance in this advice note it is unlikely that it will do any harm. However, until there is more experience of its long-term effects, it is recommended that nanolime be reserved for targeted areas and used by experienced conservators who can assess the likely effect of the treatment. They should record the details of application – including the precise areas treated, the product used, and the number and timing of applications – as part of the conservation report, and copies should be lodged with the appropriate bodies. In that way, more information will be generated so that the effect of the treatment can be more accurately evaluated. A *pro forma* record sheet for recording nanolime application is included in the Appendix.

It is intended that the information included in this advice note will provide best practice for the use of nanolime and will therefore optimise the results obtained.

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7 Appendix

Recording Treatment with Nanolime

Notes

Not all measurements and observations will be possible for each site but please complete as much as possible.

1. Depth of carbonation can be measured by application of phenolphthalein indicator to freshly drilled core sample

2. Colour change should be marked as follows:

- 2	Much lighter than untreated stonework
-1	Slightly lighter than untreated stonework
0	Same colour as untreated stonework
+1	Slightly darker than untreated stonework
+2	Much darker than untreated stonework

3. Biological growth should be marked as follows:

-1	Less biological growth than untreated stonework
0	Same biological growth as untreated stonework
+1	More biological growth than untreated stonework

4. Water repellency should be measured by applying a drop of water to the surface and marked as follows:

-1	Absorbs water less readily than untreated stonework
0	Absorbs water as readily as untreated stonework
+1	Absorbs water more readily than untreated stonework

5. Powdering should be measured by either tape test (applying tape to surface and then removing it to see how much material is stuck to it) or drawing a finger lightly across the surface:

-1	Less material removed than untreated stonework
0	Same amount of material removed as untreated stonework
+1	More material removed than untreated stonework

6. This record should be integrated into the conservation record, and copies lodged with the client and appropriate bodies, such as the architect, local authority, grant-giving body or Historic England.

Nanolime Treatment Record Form

Site	Name of site:	Address:

Stone	Location:		
	Type of stone:	Approximate dimensions (in mm): Height: Width: Depth:	Photo reference:

Condition	Surface condition:	
	General environment:	
	Colour:	Moisture level (gravimetric analysis):
	Drill resistance (include rotational speed/penetration rate and graph):	
	Permeability (drop test/Karsten tube):	

	Type of nanolime	T (°C)	RH (%)	Amount used	Method of application	Date of treatment
Treatment	Application 1:					
	Application 2:					
	Application 3:					
	Subsequent applications:					

Result of Treatment	Surface condition:			Photo reference:
	Drill resistance (include rotational speed/penetration rate and graph):			
	Permeability (drop test/Karsten tube):		Depth of carbonation:	
	Colour change:	Biological growth:	Water repellency:	Powdering:
	Other observations:		Date inspected:	
			Inspection carried out by:	

8 Where to Get Advice

Contact Historic England

East Midlands
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9 Acknowledgements

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Images

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