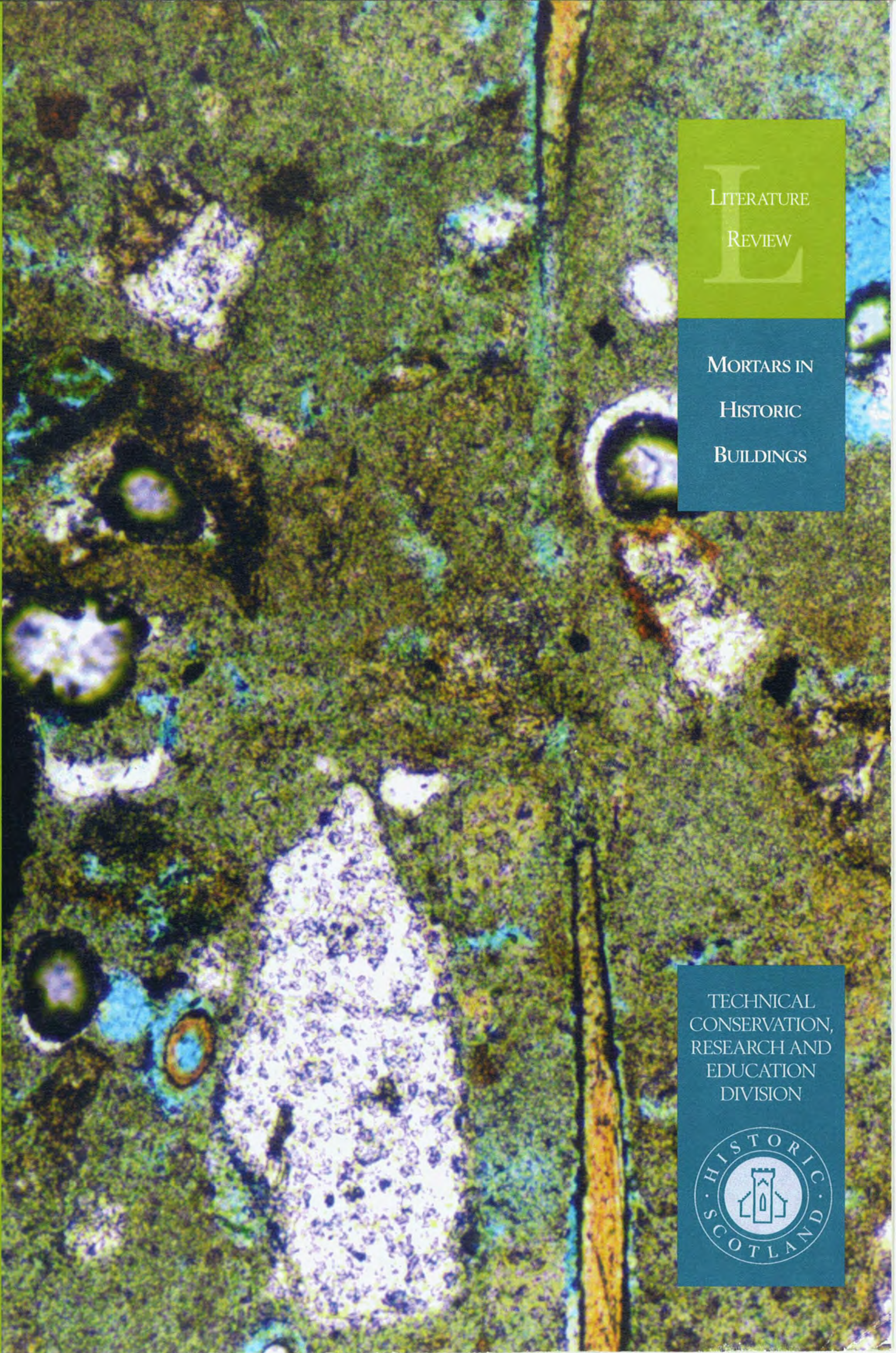
LITERATURE REVIEW

MORTARS IN HISTORIC BUILDINGS



LITERATURE REVIEW

MORTARS IN HISTORIC BUILDINGS

TECHNICAL
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HISTORIC SCOTLAND

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Literature Review

Mortars in Historic Buildings

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LITERATURE

REVIEW

MORTARS IN HISTORIC BUILDINGS.

A REVIEW OF THE CONSERVATION, TECHNICAL AND SCIENTIFIC LITERATURE

by
John J Hughes
and Jan Válek

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Note: This Literature Review covers documentation available up to May 2000

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CONTENTS

1 INTRODUCTION

CONTEXT OF REPORT - NEED FOR CONSERVATION AND COMPATIBILITY REQUIREMENTS

This review document is intended to synthesise published information regarding research carried out into mortar materials, including bedding mortars, repointing mortars, renders and grouts as found in historic buildings, but excluding clay or earth based materials. It will concentrate on masonry binders produced by the calcination (burning) of limestone and impurities, either natural or artificially added, that produce non-hydraulic and hydraulic lime based binders, natural cements, Roman cements and modern Portland Cements. However, it is also not intended to condense all the possible available information in one exhaustive book, but to select and organise a bibliography to cover the major subdivisions of this developing subject. Additionally this review will include work published predominantly in English.

The need for this review grew from the increasing interest paid to the study of mortars in historic buildings, both ancient mortars, original to the building fabric and their subsequent replacements. This not only covers traditional masonry binders such as non-hydraulic lime mortars, but also many hydraulic and artificial cements produced during the past two hundred years. As the built heritage of the past two centuries becomes increasingly valued in its own right the need for the conservation and understanding of a variety of binder, render and grouting materials has surfaced (e.g. Kirst *et al.* 1999). Increasing sophistication in conservation, mirrored by increasing technical understanding of traditional, and other modern man-made building materials has resulted in greater demands for better performance of the materials used in conservation and restoration, and their compatibility with the historic originals. Technical advances in analysis of old materials and in the production and testing of new replacements promises the possibility of meeting these demands.

The emphasis in this document is on the analysis of the demands of building conservation activities, their relationship with and influence on the choice and application of technical and scientific measures needed to fulfil these demands.

In the following sections mortars as used in historic buildings are placed in their proper context historically and technologically. Following this the concept of compatibility is introduced, as the driving force behind building conservation, and the steps currently taken by researchers and practitioners in the analysis of historic materials and the formulation and testing of their contemporary replacements. The third section will begin to review this analysis, firstly of historic mortar materials, from a conservation/practitioner angle and then from a technical point of view. The final section will cover the formulation of new replacement materials, and their testing and analysis, and consider to what extent this activity is indeed informed by the analysis of historic buildings and their original materials and a search for effective compatibility of replacements with original fabric.

1.1. Manufacturing of lime based and cementitious binders.

The vast majority of binders in historic buildings are made from lime. Historic Scotland's Technical Advice Note No.1 "Preparation and Use of Lime Mortars" (Gibbons 1995) states that lime is produced by calcining or "burning" limestone, and that lime mortars are made by "mixing lime with sand or some other form of aggregate".

The basic processes of production and use of lime can be summarised in the "lime cycle" (Figure 1). Lime can refer to different related materials, for example materials with different chemical compositions. In the production of lime a source of Calcium Carbonate ($CaCO_3$) in limestone, chalk, marble, marl, shells or coral, is calcined, (raised to an elevated temperature below its melting point) for a time long enough for the disassociation of Carbon Dioxide from the mineral lattice. This process takes place in calcium carbonate at temperatures over approximately 890°C (Boynton 1980). Calcium Oxide (CaO) or "Quicklime", is then produced which can be mixed with water, or "slaked" to form a workable putty composed of Calcium Hydroxide ($Ca(OH)_2$, or Portlandite). This mass can then be mixed with an aggregate to make a mortar which can be used for building. The mortar will then dry out and harden followed by a longer period of hardening and strength gain through the absorption of CO_2 from the atmosphere, which returns the material to Calcium Carbonate, this time not in a limestone, or other natural raw material, but in a mortar.

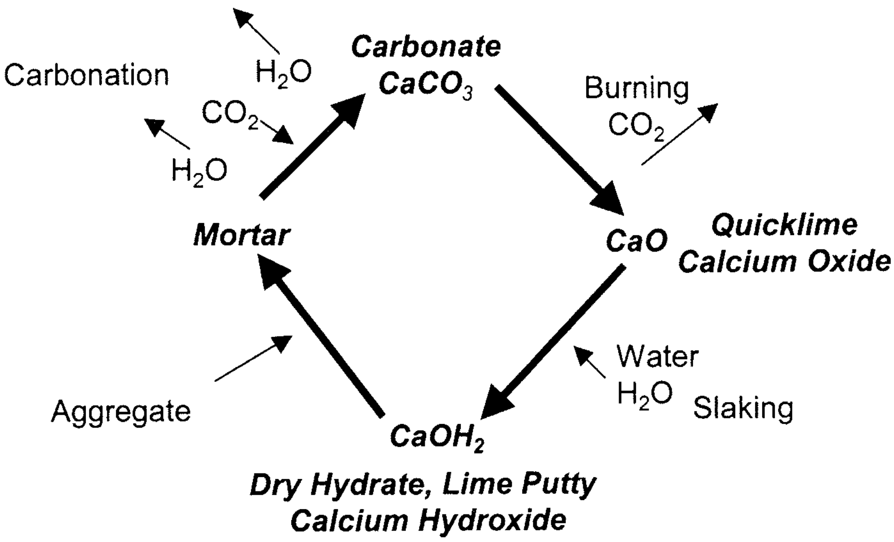


Figure 1 Lime Cycle for non-hydraulic or high-calcium lime (based on Gibbons 1995).

This apparently simple process is in practice a complex one, as variations in the composition of the raw materials and conditions and methods of application can alter the behaviour of the materials. Limestones vary considerably in composition, and are not often pure Calcium Carbonate. They can contain what are sometimes referred to, inaccurately, as "impurities", that is minerals of other compositions, most commonly Magnesium Carbonates (Magnesite ($MgCO_3$) and Dolomite Ca, Mg (CO_3)), and various silicates (mineral compounds containing SiO_4), most often clays.

The properties of lime will vary depending on the type and amount of compounds other than Calcium Carbonate in the raw material, but also on the temperature and length of burning. There are two basic types of lime used in building, non-hydraulic and hydraulic. Non-hydraulic limes are produced when limestone is used that is nearly pure Calcium Carbonate (at least 85% BS 890:1995), and can be referred to also as High Calcium Lime. Their production is typified by the lime cycle in Figure 1.

Hydraulic limes on the other hand are made from carbonate sources that contain a significant amount of silicate "impurities". These can be referred to as "Argillaceous" limestones, that contain quantities of clay. On firing within a kiln, a more complex set of chemical reactions take place that produce "hydraulic" compounds that give the material the ability to set in wet conditions (Gibbons 1995) or under water entirely. In addition to the formation of lime (CaO), the compounds that form are Calcium, Aluminium and Iron Silicates (C_3S Alite, βC_2S Belite, αC_2S Felite, C_6A_2F- C_6AF_2 Celite). These are also the main constituents of Portland Cement, and react with water to generate a chemical set. The degree to which each of these components is formed depends on the original composition of the raw materials and on the temperature and length of time of kiln firing (Ashurst 1998).

The controls on the formation of hydraulic components are well understood, due to the huge interest in the properties of cement and the large amount of research done over the past 100 years (e.g. Hewlett 1998, Soroka 1979, Taylor 1990). In cement production temperatures of up to 1450°C are used. Sintering takes place at this temperature and a recrystallised clinker is formed which is ground to form a usable powder. In addition, the raw materials are finely ground and well mixed in a slurry before firing or used direct as a dry powder.

Traditional lime production methods employ maximum temperatures of around 1100°C, and often lower. The raw materials are also not ground to ensure thorough mixing of calcareous and siliceous components, meaning that the natural distribution and type of silica and carbonate are important in determining the final composition of the lime. The lower temperatures used in lime production favour the formation of certain hydraulic compounds over others, particularly Belite (C_2S forms <1250°C) over Alite (C_3S forms >1250°C). This means that even if a hydraulic lime has the same bulk chemical composition as Portland Cement it would behave differently, because Belite hydrates and gains strength differently from Alite. The time of burning is also important in determining the nature of a hydraulic lime. The St. Astier company in France currently produce three types of hydraulic lime from a single stone type, the difference being controlled by lengthening the burning time and not the temperature reached.

The relationships between the chemistry of the raw materials and the temperature of production are described by chemists in "phase diagrams". These record the characteristics of chemical reactions and the stability of different compounds dependant on the composition and temperature and allow the prediction of what will be formed from processing materials through a kiln. The phase chemistry is well covered in standard texts on Cement Chemistry (e.g. Hewlett 1998, Taylor 1990) and will not be considered further here.

It is possible therefore to produce a wide range of materials with differing degrees of hydraulicity. These are classified as feebly, moderately and eminently, depending on the content of hydraulic components, their speed of set and eventual strength. There is a fundamental, continuous compositional variation running from pure non-hydraulic high calcium limes to Portland Cement. Historically, the use of local limestone resources that vary compositionally on a regional scale will have resulted in a variety of lime types being in use, with each area having its own lime type. Overall, traditionally produced historic materials will be more variable than modern materials, from place to place related to raw material characteristics.

Modern industrial high calcium lime production operates at temperatures of around 1250°C to reduce calcining times. Current production in the UK is almost entirely non-hydraulic, high calcium lime, made not for construction, but for the demands of the chemical industry. This material is very consistent and produced using large scale processes and very efficient kilns. Boynton (1980) and Oates (1998) give a very thorough treatment of limestone extraction, processing and calcination, particularly on an industrial scale.

1.2. History and context of mortars in buildings

When considering mortars in historic buildings it is useful to have an understanding of the history of their compositional development and use. On approach to a building of known age, an early expectation of the nature of the mortars can be formulated, and tested once the mortars are examined. Conversely, if the building is not well understood, the type of mortar may help to constrain its age. In general however, natural hydraulic binders and modern cements are not common in structures before the 19th century, when these began to be deliberately manufactured. Most mortars will consist of a lime based binder, of a varying degree of hydraulicity related to local limestone characteristics. Gypsum mortars and additions of gypsum to lime based mortars are known (e.g. Middendorf 2001) in certain circumstances, but are not generally very common. In Scotland dry stone construction was the norm for indigenous peoples for many thousands of years and the earliest mortared buildings contained earth, used primarily as a waterproofing agent to improve internal conditions (Maxwell 2000). Lime was presumably introduced to Scotland by the Romans, almost 2000 years ago, but the technology apparently disappeared after they left, only to be reintroduced at the end of the first millennium.

This part of the introduction will outline the international history of cementitious binders as revealed by selected literature. The focus here will be on demonstrable occurrences verified by direct observation, sampling and analysis. Many of the publications discussed are archaeological in nature, and the discussion below often centres on the assumptions made and the processes used to analyse and verify material occurrences.

1.2.1. Earliest uses

In a study of Neolithic lime plasters, Gourdin and Kingery (1975) identified lime processing dating from 7000-6000BC, from sites in Anatolia, Syria, Turkestan, Sinai and Jericho. The materials examined were predominantly wall and floor plasters and ornaments. The importance of this study is that they established that at this time only very rudimentary ceramics were found associated with some of the sites and that the societies that were producing the lime were "aceramic", that is the production of lime predates the development of ceramics firing technology. The production of lime requires a larger technological expertise than for the production of gypsum plasters, needing temperatures around 850-900°C compared to 100-200°C for the formation of the hemihydrate form of gypsum plaster. The volume of material needed at Cayonu, Anatolia (6500BC), is in the order of 0.67 m^3 of recarbonated lime, requiring the calcination of 4000lbs (1816kg) of limestone. Approximately 1000 lbs. (454kg) would be required per house. They postulate that rudimentary kilns must have been employed for the production of the lime, and that this provided some stimulus for the rapid development of fired pottery.

In a later paper Kingery *et al.* (1988) identify the "invention" of lime calcination and plaster at around 12,000BC. They extended and reapplied the methodology adopted by Gourdin and Kingery (1975) in comparing microstructures of materials to identify processed carbonated lime. The oldest material, dated at 12,000BC, is an adhesive used to fix a stone blade to a wooden shaft. Other non-architectural uses of lime and some gypsum "plasters" include storage jars, coatings on pottery and other jars, plaster balls, plastered skulls and sculpture. In a cave site dated to 10,400 - 10,000BC, the remains of what is considered to be a lime burning hearth were found, composed of rounded structures with a 20cm thick layer of white porous material. Examination of this material by Scanning Electron Microscopy revealed a microstructure of limestone fragments surrounded by small calcium carbonate particles - clearly interpreted as a processed lime structure. Kingery *et al.* suggest this as the earliest example of the production of quicklime. The architectural plasters examined include floor coverings, wall plasters and one case of a bench-like structure. The floor coverings were often of a single layer, polished on the top and commonly grey or white in colour, with the exception of one from Catal Huyuk which consists of up to 50 layers of 0.5mm thick. Others can be red in colour or contain larger pebbles as aggregate. Another contains the recycled red stained fragment of a previous floor covering.

Blackman (1982) examined the occurrence of lime plasters in Anshan, Iran, dating from 3500BC. As with most of the papers already discussed he suggests that clay or earth plasters were superseded once the thermal alteration of natural materials was discovered that could produce more durable gypsum and lime. He also concurs with Gourdin and Kingery (1975), that the identification of processed plasters is difficult because they are chemically identical to the raw materials, and that closer textural analysis is required. After Gourdin and Kingery's identification of lime plaster technology at 7500BC it is not surprising that the settlement studied dated at 3500BC also shows similar evidence. Their purpose was to document the occurrence, distribution and function of the materials. The materials were classified into four forms: portable (bowls etc), decorative but non-structural (wall plasters, including coloured varieties), structural and waste products from the processing of lime and the construction process.

Some bowls were examined and found to contain Calcium Hydroxide - the hydrated form of quicklime. Blackman interprets this as "..direct evidence for burned lime plaster at Anshan". Some plasters are multi-layered and coloured with pigments, and on closer analysis Blackman identifies it as fine grained quartz and carbonate, "finely ground incompletely calcined lime plaster to which no material other than pigment has been added." He does not adequately explain why the quartz is not deliberately added as an aggregate, as we might expect in a wall plaster. Lime was not used to bed the mud bricks used to build houses, but rather a mixture of mud and straw. Lime is reserved for use in decorating and plastering interior walls and floors, where it is sometimes mixed in with mud to increase strength and moisture resistance.

On considering the social and technological implications of the evidence of lime burning at Anshan, Blackman likens lime production to contemporary activities in the area, where lime burning is a summer activity. He postulates that lime burning would have taken place only during the building season, and no stockpiles of material would have been kept, due to the difficulties of preserving the material during the winter. Blackman states that calcined and slaked lime must be kept dry, to avoid spoilage, thus preventing stockpiling. This reveals some lack of understanding of the real nature of the material, especially as once slaked, even to a dry hydrate, quicklime can hardly be spoiled further. It also shows a tendency for the Archaeological writer to interpret past events with reference to current practice. Blackman concludes by stating that full time lime burning specialists were not needed. However, as lime burning appears associated with ceramic production it is reasonable to suggest that these activities were performed by the same people who were experienced in pyrotechnology.

Lucas (1948, first published in 1926), in an oft quoted description of the nature of the mortars used in Egypt prior to the Ptolomaic period (before 285BC) states quite categorically that there is no "..instance of the use of lime mortar in Egypt, or of lime in any form .. known to the author.." The mortars and plasters analysed by Lucas are composed of Gypsum, which is commonly found as a cushion between large dressed blocks of stone. Wall plasters were of the same composition and can be coloured depending on their constituents. He also states that the finishing coats of plaster can contain a large proportion of calcium carbonate and very little gypsum. This could be "poor quality" gypsum, a deliberate mixture of gypsum and calcium carbonate to produce a white colour or material processed from the naturally occurring gypsum deposits which often contain a significant proportion of carbonate. Occasionally it is just a "whitewash" that contains no gypsum at all. It is surprising that Lucas could not accept the possibility that this calcium carbonate whitewash could be lime, and that lime and gypsum plasters could have been mixed to alter the properties of the material.

Samples of Egyptian Plaster from Timna (dated 1400-1200BC) were identified by Gourdin and Kingery (op. cit.) as lime plasters mixed with quartz sand in a 1:1 ratio. This discovery contradicts Lucas's (1948) assertion that only gypsum was known before 285BC in Egypt. Gourdin and Kingery (1975) also refer to other analysis that shows lime was used in the Cheops Pyramid as well.

Gourdin and Kingery's (1975) work is also interesting in the context of historic mortar analysis. They take an analogous approach to the identification of historic processed gypsum and lime materials and their separation from natural limestone and gypsum deposits. This is important because the natural and processed materials are chemically indistinguishable. They conclude that Lucas's examination was only cursory and his simple analysis was insufficient. They visually compared the microstructures of processed gypsum and lime with their natural counterparts and use this to positively identify the matrix of several samples as being of recarbonated lime. Differential thermal analysis (see section 4.4.6) was also applied to samples of lime plaster and natural limestone. A displacement of the peaks was found between the materials which suggested that calcined and recarbonated lime can be distinguished in this way from its natural raw material. Mixtures of the two produced an intermediate peak position. However, they did not speculate on the general applicability of this method in distinguishing between natural carbonate and recarbonated lime.

In probably the earliest paper on the analysis of historic mortar, Wallace (1865) looked at the composition of mortars from Egypt, Cyprus, Greece and Rome ageing from 3000 to 1600 years old. The Egyptian mortar, from the Pyramid of Cheops was found to be of gypsum, in support of Lucas's later claims. The other mortars were found to be of lime, and the Roman mortar to contain pozzolana and a large quantity of silicic acid. Wallace concludes that lime in mortars of this age are completely carbonated and do not form a mixture of $Ca(OH)_2$ and $CaCO_3$, and that where they have been exposed, weathered or especially kept wet during hardening, that alkali-silicate will form, which may confer additional durability and hardness. Despite Lucas's (1948) assertions, lime was being used contemporaneously with construction in ancient Egypt.

Klemm & Klemm (1990) explicitly challenged Lucas's version of mortar occurrence in the old Kingdom of Egypt before the Roman Occupation, finding lime used alongside gypsum in mortars from early structures, though the pattern does vary. They also consider one of Lucas' assertions that lime was not used due to a scarcity of fuel to burn at the higher temperatures required, near 900°C compared to 450°C for gypsum. They also detected a very fine quartz intergrown with carbonates in some mortars dating to around 2490 BC. These could be interpreted as decomposition products of the hydraulic mineral C_2S, however this is uncertain, but indicates the possibility that the ancient Egyptians could have discovered and controlled the production of hydraulic lime mortars.

During the late 1980's a controversy developed over a theory that the Pyramids in Egypt were actually constructed from a form of "geopolymer" concrete. This was put forward by a respected cement chemist J.Davidovits (e.g. 1987). However, this attracted a considerable amount of criticism and has been the subject of detailed criticism (Folk and Campbell 1992).

1.2.2. Classical Greek and Roman Occurrences

It is generally held that the Greeks began the large scale use of lime-based mortars in Europe and it was from there that the technology spread to Rome (Davey 1961). Evidence exists for Greek use of lime in hydraulic works, for example a 7th century BC water channel at Olympia, and for the lining of aqueducts and cisterns (Dix 1982). In particular, the Greeks, and especially the Romans, are credited with the development of hydraulic mortars, through the use of mortar admixtures, usually volcanic ash, crushed brick or pottery, that resulted in a faster and stronger set and also made the mortars resistant to the action of water. However, there is evidence that earlier societies, such as the Phoenicians (Baronio *et al.* 1996) and the Minoans (Blezard 1998), knew of these effects.

Conophagos (1982) presents an example of early sophisticated use of lime mortars in Ancient Greece around 500BC. Mortar was used as a waterproofing for cisterns and other ore enrichment facilities in an ore smelting plant, that produced silver and lead. Two layers of mortar were used: the first being conventional, that used poor ore and tailings from the washing plant as aggregate. The second layer comprised very thin layers of a hydraulic plaster. Analysis of this shows a very high content of lead, manganese and zinc oxides and XRD analysis showed a diffuse spectrum indicating the presence of glassy material. It appears that lead oxide was added to the mortar in quantities of approximately 20-30%. The mortar without the waterproofing has normal permeability but the thin outer layer has zero permeability. Further lab experiments found that this mortar could be made by first pouring molten lead oxide into water, producing a glass, followed by powdering and addition to lime plaster. This indicates an early advanced understanding of the relationship between material properties and function in the Ancient world.

However, it was the Romans who developed the technology of building with burnt-lime mortars to a very high level. They were, however, ignorant of the chemistry of what they applied (Baronio *et al.* 1996, Harries 1995, Blezard 1998) but developed a sophisticated empirical understanding of the effects of production methods and mortar additives. Roman progress in construction using mortars and their most significant material, concrete, is considered by many to go hand in hand with social and economic progress as the empire grew (Harries 1995, Lechtman & Hobbs 1987).

Dix (1982) reviews the production of lime by the Romans, providing a valuable overview of the nature of different kilns in use at the time. Most were "flare" kilns where fuel and stone remained separate, and calcination of limestone was by radiant heat from a fire maintained below the stone, not intimately mixed with it as would occur later. Dix also discusses the development of concrete, or *opus caementicium*. This material was not widely used until the final centuries BC. Mortars were commonly used, but Etruscan and Greek building did not require large quantities as most buildings were carefully crafted from precisely cut stone, mortars only being used for reducing the friction between blocks and for cushioning. The hydraulic properties of mortar were perhaps first identified in the 7th Century BC, where they were used in a water channel in Olympia. Again the Greeks used lime to line aqueducts and cisterns in Sicily, where perhaps the Romans first encountered its use. Lime-based concretes were perhaps not developed until the 3rd Century BC. The earliest apparent uses, or at least the foreshadow of concrete, was in Cosa, used for rubble masonry house construction and at Pompeii, where rubble construction, again of houses, using pozzolanic volcanic sand allowed stronger walls to be erected to more than one storey.

Other buildings at Pompeii and Ostia (where housing developments sprang up) were walled in concrete from the third Century BC (Dix 1982). Early in the second century some temples had podiums of concrete and hydraulic limes were being used in linings. In the third Century BC lime mortars recommended for houses and for floors where reinforced by hydraulic additives. By the end of the second century concretes were commonly incorporated into the foundations of major temples in Rome, and from there their use extended to more public and domestic buildings. Vitruvius, however, doubted the reliability of Roman concrete, by the close of the first Century BC concrete was well established along with mortars and various kinds were in use (Dix 1982).

Lechtman and Hobbs (1987) describe how the advent of monolithic concrete construction heralded an architectural revolution, where building form became moulded or "cast to shape" rather than assembled. This paper discusses the form rather than the chronological history of concrete construction in Roman times and there is also a detailed survey of hydraulic binder chemistry and reactions. However, Roman construction was quite distinctive, none more so than in the use of concrete. This material permitted the use of relatively unskilled labour to erect large structures rapidly. Form-work was erected and concrete layered to form building masses (Harries 1995). Eventually wooden form-work gave way to brick, but the cores of walls remained composed of a lime-pozzolana concrete.

Roman concrete is a mixture of mortar and with a coarse aggregate. Sand was probably screened for size from the beginning of the Empire onwards. Sand type appears to have been important to the Romans. Vitruvius recommends the use of "pit sand", a sharp clean sand, considered better than river or sea sand. Lechtman and Hobbs (1987) contend that "pit sand" was clearly pozzolana. Concrete was placed by layering, and ramming where possible, and so was essentially a form of rubble masonry construction. Aggregate varied from rock, commonly volcanic or pozzolanic in nature, to fragments of demolished buildings, ceramic tile or crushed brick (Lechtman and Hobbs 1987).

Pozzolana is essentially volcanic ash, composed of aluminium-silicates that are reactive when in contact with lime, producing a network of hydrated calcium and aluminium silicon hydrates, that bring about a strong set in a mortar or concrete, and allow them to be placed under water. It is named after the town of Pozzuoli on the Italian coast near Naples, where it was first used in mortars and concretes for the construction of the town harbour, in the first half of the second Century BC (Lechtman and Hobbs 1987). Material was exported widely from the region for numerous harbour projects. It is in the development of harbours that the Romans gained their first experience in the handling of concrete.

Between 22-9BC King Herod built what was the largest harbour of the age at Sabastos, 45 km south of Haifa (Oleson *et al.* 1984). Artificial breakwaters were constructed of enormous concrete blocks on a bed of loose rubble, that enclosed an area of some 20 hectares. Much of the materials used were imported from as far away as Italy, including the reactive pozzolana sands. Some of the concrete blocks have a volume of up to 125m^3. The remains of sophisticated wooden form work have been found, that have hollow wall sections and no floors. They are interpreted as being floated out into position and then the hollow walls filled with mortar to sink the moulds. They were then filled with concrete and left in place.

Concrete remained a material primarily used for hydraulic works until the 1st and 2nd Centuries AD. Considerable experience had been developed in this application, but Letchman and Hobbs (op. cit.) bring into question how the Romans transferred this technology to large vaulted buildings. Form work for buildings became standardised on brick or stone, from the end of the second Century BC (Harries 1995). However, it is clear that progress in application was slow and incremental as the quality of the mortars and concretes used was not always high. By 40BC, during the reign of Julius Caesar, construction had become more reliable, based upon a better choice of materials, and their more sophisticated use. This includes the selection of aggregates depending on the building element being constructed, lighter aggregate being used for walls and denser for foundations. During the period from 120BC until Nero built his Domus Aurea in 64-68AD, which made much use of concrete, slow empirical progress took place. The paper by Letchman and Hobbs is an excellent survey of Roman concrete technology.

Harries (1995) points out that Roman aggregate of 50-150mm, could not be mixed manually, so concrete was placed in layers by hand, resulting in a higher mortar: aggregate ratio than today. This different form of construction of brick-filler-brick changed the structural stability of constructions, as they were now essentially monolithic. Brick arches were built in to strengthen vaults and left as an important element of the concrete mass. Bonding courses were also common, through-wall brick tiles every 15-25 courses. It is doubtful if they actually served this bonding purpose, being more likely to have marked the end of a day's concrete placement, or for levelling. However, the bonding courses do have the effect of preventing seismic damage, by absorbing energy through encouraging relative movement between bonded units.

Domes were the major architectural innovation of Roman Construction. Constructed over formwork, carefully graded aggregate was used from heaviest at the bottom to lightest at the top. The dome would also thin toward the top reducing the weight of the structure further. The Pantheon in Rome (128AD) is the most impressive example, and relies partly on its immense concrete ring foundations (4.5m deep by 10.3m wide) for stability in such a seismically active region.

During the late Roman era and into the Byzantine period, mortars were commonly produced with crushed brick aggregate which acted as a pozzolana. The joint thickness of brick wall facings also increased from 10mm to 70mm (Baronio *et al* 1996). Aggregate size also increased to up to 25mm in these mortars. The role of the thick mortar joints is not entirely clear but they do allow greater deformation of a structure without catastrophic damage, especially at an early age, thus allowing settlement of differential movement (Binda *et al.* 2000).

Despite the preponderance of pozzolanic mortars in Roman Construction and their hydraulic properties there is little evidence that they deliberately calcined limestone to produce *hydraulic binders*. Mishara (1982) reviews the composition of artificial Portland Cements and natural hydraulic mortars, and considers to what extent binder manufacturers in the ancient world deliberately produced hydraulic binders. She asserts that evidence for this is missing. Pozzolans were used extensively and clearly deliberately, however, documentary evidence of the time warns builders away from the use of "grey variegated" stones - those that may well have produced hydraulic binders. For example Vitruvius in 'The Ten Books on Architecture' (Morgan 1960) writing around 25BC described how to use lime and prepare mortars;

> '... , *next with regard to lime we must be careful that it is burnt from a stone which, whether soft or hard, it is in any case white. Lime made of close-grained stone of the harder sort will be good in structural parts; lime of porous stone, in stucco. After slaking it, mix your mortar, if using pit sand, in the proportions of three parts of sand to one of lime; if using river or sea-sand, mix two parts of sand with one of lime. Theses will be right proportions for the compositions of the mixture. Further, in using river or sea-sand the addition of third part composed of burnt brick, pounded up and sifted, will make your mortar of a better composition to use.*'

This work set, for that time, very important standards but also certain prejudices towards non-hydraulic and hydraulic mortars. It was believed that only the hardest limestone could be used to produce hard and durable mortars. (Sickels 1987). Portland cement manufacture depends on the formation of clinker, which needs to be ground to a powder before use. This would have been difficult in ancient times, and large lumps of clinker would not have performed very well, putting many users off.

Vitruvius advises that the "best white stone" be chosen, and advice is given to avoid mixtures of limestone and clay. Mishara suggests that the analysis of artefacts may allow us to decide what people actually knew and what influence advice like Vitruvius had. Relationships between materials and building patterns may allow a judgement of whether considered choices had been made to select a particular material for a particular job. The link with ceramic production, identified by others (above) may be very strong where the use of artificial pozzolans is concerned.

The techniques and materials of Roman construction are well researched. The publications mentioned above only cover a small fraction of the available literature. The intention in this section was to give the reader some idea of the breadth of the subject and of the basic chronological evolution of Roman mortar and concrete technologies.

1.2.3. Other world-wide occurrences

The use of lime-based mortars through history is not limited to Europe, Northern Africa and the Middle East. Malinowski (1981) notes their use over the past 2000 years in the Great Wall of China, monuments in India and in Central America.

Lime was extensively used by the Mayan civilisation in Central America, or "Mesoamerica". Littman (1957) comments that the use of lime is restricted to that area, and is a distinguishing feature that may have played a role in the development of "more advanced forms of architecture" compared to other parts of the New World. In the samples he collected from the steps of a Maya temple at Comalcalco, Mexico (dating to approximately post 200 AD). Littman identifies lime plasters, wash coats and "lime-aggregates" (monolithic lime masses). On analysis of these he found that the ratio of calcium to magnesium supported an origin for the lime in burnt-shell materials and that the different classes of materials had different, but fairly consistent insoluble residues (aggregates). The materials were also applied in a repetitive order indicating highly organised construction tailored to function.

Hansen *et al.* document occurrences of Maya plasters covering walls and floors dated to the early Middle Preclassic period (1000-300BC). The most substantial use of lime materials in Mayan architecture was for rendering surfaces and for flooring. Hanson *et al.* describe how variations in technological style of materials can be related to function and to age, indicating a high degree of sophistication in application. They analysed a group of materials from an ancient Maya site and found sorting of aggregates more prevalent in samples dated 300BC - 240AD (Late Preclassic) and also in lime plasters.

MacKinnon and May (1990) report the excavation of a Mayan lime processing site in Belize, dated 250-600AD (Early Classic), consisting of a Midden with numerous potsherds and a layer of enriched calcium carbonate, similar to those seen in current small scale lime processing operations in the same region. A similar site was identified by Mazzullo and Teal (1994) in Belize, who contended that the presence of a carbonate enriched layer in sediments was not convincing evidence for lime processing on its own. They develop the recognition of processed lime materials through the comparison of microstructures, and careful mineralogical analysis. Pottery sherds were found in a matrix composed of calcite. Analysis shows the presence of calcite, quartz and kaolinite (from the pottery) but also <2% MgO, CaO and $Mg(OH)_2$, thus supporting a processed lime origin, as the oxides are not natural materials.

The identification of lime processing by the Maya, as presented in the papers discussed above, is used in interpretations of social, technological and architectural development amongst those peoples. Large amounts of lime were required for their building programmes, but little direct evidence of their methods exists (Mazzullo and Teal, 1994). According to Hansen *et al.* (1997), the transformations in Maya society are not only visible by the built environment but also through the study of the formulations of burnt-lime building materials they used.

There is a considerable amount of further literature on Mayan lime, covering a well established area of study. The publications discussed here only serve as an introduction. As for the use of lime-based materials in other areas of the world there appears to be a paucity of research and publication activity.

1.2.4. The Renaissance to the 19th Century: the development of hydraulic mortars and cement

It is generally held that following the decline of the Roman Empire the technologies that they developed to such high levels declined, and much information and experience was lost (Blezard 1998, Callebaut 2000). Certainly the widespread use of pozzolanic sands to create hydraulic mortars largely disappeared, except in Byzantine construction (e.g. Camak *et al.* 1995, Binda *et al.* 2000). It was during the Renaissance that architects such as Alberti, Martini and Palladio followed the techniques of Vitruvius (see above) and took inspiration from classical Greek and Roman buildings (Baronio *et al.* 1996, Sickels 1987, op. cit.). They continued to seek out hard, white limestone to produce lime and the use of pozzolanic additives, either natural or brick dust.

The majority of the following discussion of the development of hydraulic binders and Portland Cement during the eighteenth and nineteenth centuries is drawn from two sources: Blezard (1998) and Sickels (1987), both excellent reviews of aspects of the history of calcareous cements and both containing extensive further documentary references.

The techniques and theories described by Vitruvius were largely followed till John Smeaton started experiments during building preparations for the Eddystone lighthouse in 1756 (Blezard 1998). He proved that regardless of colour of the limestone equally strong limes can be produced and showed which limes are able to set under water. He found that Blue Lias Lime from Aberthaw, which contained a proportion of insoluble clay, showed the best hydraulic properties, and indeed that it was the presence of this clay fraction that was necessary to produce a hydraulic lime. This was the first time that the properties of hydraulic lime were properly recognised. He also experimented with pozzolanic earth imported form Italy (Cowper 1927), and other artificial pozzolanas, including forge scales.

The major issues that were disputed from the mid-eighteenth to mid nineteenth centuries are, according to Sickels (1987):

- the chemistry of lime and the controls of hydraulicity
- the quality of mortar derived from additives
- the effect of storage of mortar on performance.

As noted above, Vitruvius stated that the strongest lime was produced from the hardest white limestone. Smeaton established that the colour of the stone was immaterial, that hydraulic limes contained clay and that to obtain hydraulic properties the materials needed to be burnt together. His research into additives also led him to believe that substances containing iron, or "ferruginous" elements, were also necessary to produce a strong lime. Others also held this view and several treatises were published on this in the late 18th Century, that also favoured manganese as a ferruginous agent. A debate ensued with two camps forming, one favouring clay as the cause of hydraulicity and the other favouring ferrugenous compounds.

The clay supporters ultimately won, when workers such as Vicat, Descotils, Treussart and John began experimentation. Vicat was the leader, and his work soon confirmed Smeaton's theories, but he also identified the presence of aluminium and silica in the clays, that were required before hydraulic materials could be formed. He also developed the first test for the setting behaviour of a mortar, and experimented continually to determine the role of clays and magnesia in the hydraulic characteristics of limes. He divided limes according to their hydraulic qualities into five categories, which are still held by many to be valid (Vicat, translated by Smith 1837, Pasley 1838):

- Fat lime- does not set under water, can be entirely dissolved by water, doubles its amount when slaked.
- Lean lime- does not set under water but dissolves only partially, when slaked, its volume increases very little.
- Moderately hydraulic lime- set under water after 15 or 20 days, then continues to harden but very slowly.
- Hydraulic limes- set under water after 6 or 8 days, continue to harden, but the greater part of it takes place during first six months.
- Eminently hydraulic limes- set under water after 2 or 3 days, after one month, they are already very hard.

To determine the setting time, Vicat used a knitting needle, which he immersed into the sample. An improved version of this test is still in use.

The period from 1756 to 1855 was a time of considerable experimentation by many workers attempting to produce a good hydraulic mortar for the building industry. Further work was pursued by workers such as Pasley into the formulation of artificial hydraulic limes, with some success. At the end of this period, Sickels (1987) attests, that numerous new formulations of mortar had been experimented with, and that it was now possible to "know which ingredients would produce the desired effects".

A gradual evolution of hydraulic cements took place over the 19th century in many countries, including France and the USA. Vicat prepared an artificial hydraulic lime by calcining an intimate mixture of limestone and clay, ground together wet (John 1819, in Blezard 1998). There were lots of patents for different materials at this time. Joseph Aspdin was a builder and bricklayer from Leeds, who lodged the most famous of these, no. 5022 on October 21 1824, for the material described as Portland Cement. Aspdin's process took a hard limestone, crushed and calcined it, ground it to a fine wet slurry and mixed it with clay and then calcined it again, finally grinding the final product once more. He must have used a low temperature, for this material was nothing more than a hydraulic lime, and not the Portland Cement we understand today. Aspdin's son, William, left the family firm in 1843 and started his own cement works in London, where he discovered that "overburnt" or clinkered materials increased the resultant strength, though this is considered to be an accidental innovation.

Throughout this time and until the mid to late 1800's, James Parker's Roman Cement, patented in 1796, was the most successful cementitious product available. It was a natural hydraulic binder, produced from the calcination of Septarian nodules from the Isle of Sheppy. After this patent expired in 1810, several new products rushed on to the market, ultimately leading to the development of artificial cements by the likes of Atkinson, Frost and Aspdin. Parker believed that his product was superior to others due to the temperature of production, which is directly related to the degree of hydraulicity developed in a cement, and as such he was the first to link temperature of production with setting and strength development. Most hydraulic lime production at the time was underburnt, not reaching a vitrified state, but Parker took his materials to barely vitrified. Parker also ground his material, and instituted quality control on every barrel sold, by testing the setting time of a small sample of each. If it didn't set in 20 minutes then it was rejected (Sickels 1987). The competition carried on underburning until William Aspdin's accidental development of vitrification.

I.C. Johnston also discovered that overburnt material produced a stronger, though slower, setting cement, and it was he who fully appreciated the need for vitrification in burning raw materials (Davey 1961). Johnston is considered to be the real originator of Portland Cement as we understand it today. There was a very competitive industry at this time, though it appears that Aspdin's original patent was merely for a proto-portland cement, and it was only after his son and later I.C. Johnston in the mid 1840s synthesised a vitrified binder at higher temperatures.

According to Blezard (1998) the development of Portland Cement during the 19th and early 20th Centuries follows a path through proto, meso and normal Portland Cement. They are characterised by increasing temperature of production, increasing reaction between silica and calcium, increasing strength and increasing control and uniformity of manufacture. The development of the rotary kiln around 1878 (Davey 1962, Bye 1983) is perhaps the most critical step in the production of modern cements, resulting in a high degree of controllability and uniformity of product. From then on, the higher temperatures used for production produced a very fast setting cement, so from 1890 Gypsum has been added in a small quantity to all Portland Cement as a set retarder. Since then, in modern building, cement based mortars and concretes gradually replaced the lime-based mortars.

After the invention of Portland cement, research has concentrated mainly on its development and a new, 'modern' way of building has begun. Cement mortars were considered better, stronger, more durable and with a more reliable hardening process than lime-based mortars, which were soon superseded by the cement gauged or pure cement mortars. Approximately from the First World War onwards cement rich mortars and renders were used in the repair and restoration of historic buildings (Burman 1998), where they had most often not been part of the original fabric. It was only after serious failures, where inappropriate use of the cement mortars apparently damaged the valuable original masonry, that a growing interest in lime-based mortars reappeared. However, the advent of Portland cement as the dominant mortar binder during the 20th Century, resulted in all research efforts being focussed away from lime-based mortars, and particularly its use in building conservation.

This was redressed partly by events like the publication of the Venice Charter (1964), that was approved during the 2nd International Congress of Architects and Technicians of Historic Monuments held in Venice that year. Although it did not affect directly the use of mortars, it set out conservation and restoration principles that supported scientific research to underpin the better conservation of the architectural heritage.

2 DEFINITIONS AND FUNCTIONS OF MORTAR IN HISTORIC BUILDINGS

The use of mortars (bedding, rendering, grouting etc) determines their function (structural, protective, decorative etc.) within the structure. The mortars are required to possess certain properties imposed by their use. Careful consideration of the use, functions, classifications and materials used in mortars of historic buildings is very important for the effective analysis of historic mortar properties as well as for the specifications of modern mortars. The way in which a definition is expressed forms and limits the understanding of the defined material. Therefore, definitions vary according to their purposes. Definitions in standards (e.g. British Standard) are usually limited to a minimal description of the composition of a material. Next to these basic standard definitions there are definitions incorporating correct use and desired property of the material. Such detailed definitions are often needed in practice to provide further explanation according to a particular area of study.

The aim of this short comparative study is a discussion of basic definitions related to mortars and masonry conservation. On the other hand it is not to form new precise definitions or to list and cover all definitions related to this subject. The key terms were already defined and published in special technical dictionaries and standards (e.g. Walker 1988, BS 6100: Glossary of building and civil engineering terms) or as a terminology of scientific and technical literature (e.g. Holmes and Wingate 1997, Sickels 1987). These publications should be referred to for a full list of terms and definitions.

2.1. Key terms definitions

Definitions of the most relevant terms such as 'mortar', 'concrete', 'binder', 'cement' and 'aggregate' are discussed and often more than one version of the definition is presented here to cover all aspects. The intention is to review all these viewpoints. It also aims to highlight discrepancies between terms caused by their different comprehension in different contexts. For example, Goins (2000) pointed out that the term 'mortar' is ambiguous as it means *a compound that holds blocks of masonry* as well as having a broader meaning of *any historic cement and aggregate*.

2.1.1. Mortar

The analysis of various definitions of mortar illustrates their interrelation with their purpose. The most general definition is stated by British Standard (BS 6100 part 6.6.1:1992 Building and civil engineering terms). It defines mortar as:

'Mixture of binder, fine aggregate and water that hardens.'

The definition states the general mortar composition and process of hardening but it leaves out the use, functions and properties. For building practice, however, there may exist a need to extend this definition and express additional requirements. Holmes and Wingate (1997) define mortar from a more practical point of view as:

'Any material in a plastic state which can be trowelled, becomes hard in place, and which can be used for bedding and jointing masonry units.'

In this definition the main objective is on the requirement for workability (albeit trowelled is not a very exact requirement) and description of the use of mortar. It does not specify any material requirements. On the other hand, a definition presented by Goins (2000) is more explicit about the composition of mortar. It states that mortar is:

"A pasty substance formed normally by the mixing of cement, sand and water, or cement, lime, sand and water in varying proportions. Used normally for the binding of brickwork or masonry," from Cambridge Dictionary of Science and Technology (Walker 1988).

The RILEM Technical Committee-167 COM 'Characterisation of old mortars, with respect to their repair' discussed a detailed definition which would explicitly express even quality requirements for the mortar. It states that:

'Mortar is a mix of organic and inorganic binders, mainly fine aggregates, water and admixtures and organic and inorganic additives, mixed in order to give to the fresh mortar a good workability and to the hardened mortar adequate physical (porosity, vapour permeability etc.) and mechanical (strength, deformability, adhesion etc.) behaviour and good appearance and durability.'

This definition is the most relevant for mortars in historic buildings and conservation. It is based on findings from a recent research into requirements for a compatible mortar used for conservation of masonry. The words such as good workability, adequate physical and mechanical behaviour and good appearance need to be defined further according to the use and required function of the mortar.

2.1.2. Concrete

British Standard (BS 6100:6.2:1986) defines both mortar and concrete in a similar way. However, there is a distinction between them as concrete contains hydraulic binder and may or may not contain fine aggregate. The BS defines concrete as:

'Mixture of aggregate, hydraulic binder and water, that hardens.'

Holmes and Wingate (1997) use the distinction based on the size of aggregate. On the other hand, Goins (2000) pointed out that the terms 'mortar' and 'concrete' are used interchangeably. In historic structures a massive wall was often built as a multiple-leaf wall with a mortar/concrete infill containing a larger aggregate and stones. Strictly by definition, it should be called concrete, however, the word concrete is more associated with Portland cement. For historic structures where lime based mortar was used it is more common to call it mortar. Terms such as 'Roman mortar', 'Roman concrete' and/or 'natural concrete' are also terms which are used to describe infill mortar based on hydraulic binders. Any misconception should be avoided in the case of concrete made from modern Portland cement.

Kumar Mehta and Monteiro (1993) define concrete as:

'A composite material that consists essentially of a binding medium within which are embedded particles of fragments of aggregates.'

Their definition describes concrete from a structural and functional point of view rather than by its composition. This definition indirectly suggests something that is omitted in the previous two definitions. Unlike mortar, concrete may be used by itself to build structures. Mortar is used for jointing masonry units or for their surface coating.

Roman Mortar and Concrete

There is more than one description of 'Roman mortar' and/or 'concrete'. In general it contained lime (could also have been hydraulic), sand, a certain amount of pozzolana and often also pulverised bricks.

A number of different Roman building techniques have been described (e.g. Vitruvius). For example, Opus Caementicium is a name for a building technique used by Roman builders when 'Roman mortar/concrete was used to set undressed stones called caementa' (Sickels 1987).

2.1.3. Binders

Binder is defined in the British Standard as:

'Material used for the purpose of holding solid particles together in a coherent mass.' (BS 6100:Section 6.1).

This definition covers the most commonly used Portland cement but also lime or clay. This term is in some cases interchangeable with the term cement. Binder can be subdivided by its hydraulic nature into hydraulic binder and air hardening binder.

Lime

Lime covers a wide range of lime products such as hydrated lime, lime putty, quicklime, hydraulic lime, non-hydraulic lime (air lime) etc, in general 'all of the oxides and hydroxides of calcium and magnesium, but excludes the carbonates' (Holmes and Wingate 1997). The most fundamental distinction between building limes is the same as for binders and that is their subdivision by hydraulic nature.

Non-Hydraulic Lime, Air Lime, Air Hardening Lime

Air hardening lime is defined by prEN 1996 as:

'Lime mainly consisting of a calcium oxide or hydroxide which, when incorporated into a mortar mix, slowly hardens in air by reacting with atmospheric carbon dioxide. Generally they do not harden under water as they have no hydraulic properties.'

Hydraulic Lime

It is a hydraulic binder. 'It sets and hardens by chemical interaction with water and is capable of doing so under water.' Defined by BS 6100:Section 6.1.

Holmes and Wingate (1997) define Hydraulic limes as Class C limes.

They *'are natural hydraulic limes prepared from limestone or chalks with clay impurities. Artificial hydraulic limes are manufactured by mixing pozzolan with calcium hydroxide which enable the limes to harden even in damp conditions.'*

Hydraulic limes are classified according to their quality. Hughes & Swan (1998) pointed out some discrepancy between current systems of classifications. They explained that Vicat (reprinted edition 1997) classified hydraulic limes according to their setting times as feebly hydraulic, (moderately) hydraulic and eminently hydraulic. Eckel (1922) shortens the classification to feebly and eminently based on cementation index 0.3-0.7 and 0.7-1.1 respectively. Cementation index was also used by Boynton (1980) who divided limes into feebly (0.3-0.5), moderately (0.5-0.7) and eminently (0.7-1.1) hydraulic. On the other hand British Standard (DD ENV 459-1:1995) uses a system of classification based on compressive strength where, for example, the denomination HL 2 corresponds to the compressive strength from 2 to 5N/mm^2 at 28 days (Holmes 1998). These classification systems are contradictory for some hydraulic limes, and for conservation purposes an improved system based on actual performance would be beneficial (Hughes & Swan 1998).

Cement

'A material for uniting other materials or articles. It is generally plastic at the time of application but hardens when in place.' Cement by this definition (Walker 1988) includes all lime mortars and Portland cements (Goins 2000).

As defined above, the terms cement and binder can be used interchangeably. However, in the context of traditional building material, cement is defined by Holmes and Wingate (1997) as:

'Quick-setting binder for making mortars and concretes. By far the most widespread cement is the Portland cement formed by grinding a clinker which has been prepared at high kiln temperatures from a mixture of clay and limestone. There are, however, other forms of cement including natural cements formed from naturally occurring nodules of calcareous clay (such as Septaria). A distinction between these and other hydraulic limes is that cements must be ground to a fine powder before they can slake'.

In this context, cement is considered as quick setting hydraulic binder. In the building industry the term 'cement' is understood to mean Portland cement or mortar made of Portland cement. The term 'cement' in its meaning as a general binder is not used in the building industry.

Portland Cement (PC), Ordinary Portland Cement (OPC)

Holmes and Wingate (1997) define Portland cement as:

'Common form of cement conforming to certain standards and made by grinding a clinker formed by firing a slurried mixture of clay and limestone at high temperature in a kiln. Calcium sulphate is also ground in to modify the setting rate.'

Natural Cement

The distinction between natural cement and Portland cement is expressed in the definition of cement by Holmes and Wingate (1997). Goins (2000) defines natural cement simply by its composition and stresses that it is a natural mixture:

'Made by calcining natural mixtures of calcareous and argillaceous materials. Examples are Roman concrete made of the natural cement and eminently hydraulic lime.'

Pozzolan, Pozzolanic Material

Holmes and Wingate (1997) defined pozzolan as the following:

'Pozzolan is any material which contains constituents, generally aluminia and reactive silica, which will combine with hydrated lime at normal temperatures in the presence of moisture to form stable insoluble compounds with binding properties.'

Pozzolan possesses by itself little or no cementitious value and could be regarded as an aggregate, however, when it chemically reacts it gains certain cementitious properties (e.g. crushed bricks or brick dust added to lime based mortar).

2.1.4. Aggregate

Holmes and Wingate (1997) define aggregate as:

'the hard filler materials, such as sand and stones, in mortars, plasters, renders and concretes.'

Or, alternatively, it is defined by Sickels (1987) as:

'any granular material, such as sand, gravel, crushed stone, or iron blast-furnance slag, used with a cementing medium to form a mortar. It is usually the largest volumetric constituent of a mortar.'

In general, an aggregate can be regarded as any solid material apart from cement (binder) and reinforcement added to a mix immediately before or after mixing. Fibres and other materials added to a mix, which are sometimes called reinforcement but do not act as the reinforcing agent, should be regarded as an aggregate. Similar un-reacted pozzolan, un-burnt and/or un-slaked lime lumps originally meant as cementing materials act as an aggregate. For example, when analysing binder:aggregate ratio of lime mortars containing lime lumps (un-burnt and/or un-slaked lime) by acid dissolution the effective binder content is less than the actual figure derived (Leslie and Gibbons 2000).

Sand

Sand is a fine aggregate usually referred to by a specific name after its origin or nature. Holmes and Wingate (1997) define it as:

'weathered particles of rocks, usually high in silica, smaller than gravels and larger than silts, typically between about 0.06 mm to 5 mm. The particles are hard and will not crumble.'

2.2. Classification of mortars

In modern building practice the classification of mortars provides their simplified description according to the purpose of their application. When dealing with conservation of historic structures incorporating mortars, tasks such as analysis of historic mortars and design of new repair mortars should be undertaken. The classification helps to describe the historic mortars and find new mortars. Analysis of historic mortars aims to describe mortar and its properties (appearance, composition etc). The properties of mortar are primarily dependent on its binder, hence the classification according to the binder is one of the basic distinctions between mortars.

Another aspect of the classification is that a mortar has its particular function. This function imposes certain requirements on workability and properties of a hardened mortar. The design of a new mortar is based on matching its properties with these requirements. The main differentiation of mortars in this context, therefore, is based on the nature of binder and the purpose of their application in relation to the function of mortar within the historic structure.

2.2.1. Historic and modern mortar, original and new mortar

These are often vague terms, which are used in literature to distinguish between mortars present in the historic structure before and after any contemporary conservation work when a new mix was introduced. The term 'historic mortar' can be used to denominate the original mortar but also the mortars used later for repairs. A modern and/or new mortar is understood to be a mortar used recently in conservation works.

Although these terms are not material-related, historic mortars are often presumed to be lime-based mortars. The reason for this is that within European architecture, the majority of historic buildings were made of stone or brick masonry with lime based mortar. Non-hydraulic lime mortar was the most common mineral binder throughout architectural history (Furlan 1991). Hydraulic limes and mortars with hydraulic properties were increasingly specified from the seventeenth century onwards (Lynch 1998) and later, at the end of the 19th century, cement based mortars began superseding all the lime based mortars. However, other historic mortars based on clay or mud were not uncommon. Some historic mortars based on non-hydraulic lime mortar had hydraulic properties from additives like pozzolan, volcanic ash and/or crushed bricks.

There is also confusion regarding the term 'historic building', which is sometimes used as a general denomination of protected or listed structures. However, such historic buildings can still be made of a relatively modern material, e.g. a mortar based on Portland cement. Relatively recent historic buildings may be made of an early reinforced concrete. Watt and Swallow (1996) wrote that 'age is just one of the factors that makes a building "historic", along with architectural and historical association with important people and events'.

2.2.2. Distinction based on the nature of binder

The most common distinction between mortars is based on the nature of binder. Binder can be non-hydraulic or possess various degrees of hydraulicity. The hydraulic properties of the binder affect the physical and chemical properties of mortars (e.g. strength, hardening under water, solubility and reactivity of the binder, formation salts etc.). Subsequent to this basic distinction, each mortar based on a particular binder can be subdivided further according to various criteria (e.g. quality, composition, aggregate etc). A common way of subdivision is a description of a mortar product for marketing. For example, in the case of lime mortars, Wingate (1992) suggests that a good description should contain the type of carbonate from which the lime was made, the form in which the lime is traded and the quality of the product.

Distinction based on the nature of the binder
• Mortar based on Portland cement
• Mortar based on clay/mud
• Mortar based on several binders
• Mortar based on lime
• Mortar based on gypsum
• Mortar based on organic/synthetic binder

Table 2.1: Classification of mortars based on the nature of binder

Synthetic binders are not normally recommended for repair of historic structures. However, 'organic' mortars or mortars with organic additives were often used in the past (Sickels 1981). The most common were eggs and blood (Sickels 1987). Natural organic additives are susceptible to microbiological decay and a modern substitution is desirable (Herm 1993).

Synthetic additives are relatively new to conservation and can be designed to match the properties of the organic additives. Mortars modified with the synthetic/organic additives can possess particular properties and are tested for specialised conservation of frescos and wall paintings (Michoinova 2000). Synthetic additives are also used as water reducers (Torraca 1995).

2.2.3. Distinction based on the purpose of application

Mortar in historic structures functions in many different ways as plasters (renders) on internal and external walls, supporting substrates for frescoes, bedding mortar of masonry, supporting material for pavements and mosaics, and watertight lining materials in cisterns, wells, aqueducts etc., (Moropoulou *et al.* 2000). Today's distinction suggested by the RILEM technical committee (TC-167COM, unpublished discussion document) is as follows:

- Mortar for external and internal coating (render, plaster, harling etc)
- Masonry mortar (bedding, pointing/repointing, core filling mortar)
- Mortar (adhesive mortar) for fixing architectural details (e.g. tiles, cladding, floor panels)
- Mortar for special use (e.g. watertight lining)

In conservation practice the use of mortars sets down their properties. Therefore, fresh and/or hardened mortars vary in composition and workability according to their use. Conversely, properties of historic mortars can be deduced from their use and function. In conservation, mortars can be further divided according to their application.

Mortars can be used in the following conservation techniques:
• Pointing, repointing
• Rough racking
• Tamping
• Bedding, replacement of masonry units
• Internal and external coating – plasters, renders and their repair
• Plastic repair
• Injection, grouting

Table 2.2:This distinction is based on conservation techniques described by Ashurst (1983) in 'Mortars, Plasters and Renders in Conservation'. In this book, the author describes the practical use and preparation of mortars.

2.3. Repair techniques and specific functions of mortars used in conservation

Mortars can be divided according to their use (as discussed above). In this chapter mortars used for individual conservation techniques are discussed.

Profound understanding of the use, function and composition of mortar is crucial in order to repair historic masonry or to design a new mortar compatible with it. Moreover, in the conservation of historic construction every task requires a different solution to achieve the desired result. Understanding of material interaction with other materials and environments is much more significant for materials used in conservation than for materials used in the modern building industry. The materials and techniques used in conservation must meet special requirements such as compatibility, reversibility, re-treatability etc.

For example, mortars are required to be weatherproof but breathable, and the mortar should not inhibit moisture evaporation from the masonry. In some special cases the mortar is used as a sacrificial layer (a protection of a substrate where the mortar is designed to deteriorate). Portland cement mortars are not suitable for remedial works on lime-based masonry as they are impermeable, possess low porosity, can become a source of soluble salts etc. (Feilden 1998). On the other hand, Von Konow (1997) suggests that lime-based mortars with a wrong composition can cause damage as well. Non-hydraulic lime mortar may cause an accelerated decay, for example in conjunction with sandstone (Maxwell 1994).

Special repair techniques use special mortar mixes, which should be designed to be compatible with the original masonry. However, the repair technique itself should be compatible and the repair should not cause any damage to the masonry during the application or in the future. For example, Binda *et al.* (2000) pointed out that repair techniques such as grouting, wall/pier jacketing, concrete ties, roof and floor substitutions could be inappropriate in seismic areas where the repair can multiply the damage caused by earthquake. The repair techniques should respect the original material and structures otherwise they become incompatible (Binda *et al.* 2000).

2.3.1. Pointing, repointing

Pointing is a basic masonry practice, to fill and finish the joints with mortar after the stones are laid down into bedding mortar. The type of pointing originally depended on the masonry character and various styles were recognised in Scotland, e.g. flush pointing, smear pointing etc. (Maxwell 1998). The thickness of the joint can also be related to the selected pointing tools and their width (Maxwell 2000). Therefore, repair and repointing work should be based on a recognition of the diversity in styles and the various traditional building techniques (Maxwell 1998). In ashlar masonry the pointing appears as a very fine white joint (usually pure lime putty was used). However, the construction involves a joint of a wedge shape as the ashlar blocks are undercut back from the face of the stone to allow a better adjustment of the blocks (Davey *et al.*, 'The Care and Conservation of Georgian Houses' 1979).

Pointing (repointing) has, in principle, two effects. It forms the appearance of the masonry by its colour and type. It determines the durability of the masonry by improving its resistance to weather exposure. Deterioration of pointing mortar in joints can be seen as a first sign of danger for the whole masonry as the mortar does not seal the masonry sufficiently against driven rain water etc. Therefore, to preserve and prolong the durability of masonry any open joints without mortar should be refilled/ repointed.

Repointing has to be carried out with special care since it can affect both the durability and appearance of the masonry (Maxwell 1998). Pinnings, small stones (e.g. fragments of the local stone used for masonry, slate, oyster shells, bricks, tiles) were originally part of rubble masonry and played an important part in the appearance of the surface finish and pointing. Pinnings also functioned structurally in connection with the wall construction technology (Gibbons TAN 1 1995). Pinnings often significantly reduced the visible proportion of mortar. Depending on the character of the masonry and surface finishing technique (smear pointing etc.), pinnings were either visible or hidden. In current practice, pinnings are often used to minimise drying shrinkage by reducing the proportion of lime mortar in masonry, and helping the carbonation process (Gibbons TAN 1 1995) (permeable stone should be used). If the character of masonry is to be preserved, pinnings should be kept or replaced in joints during the repointing work.

Another reason for repointing historic masonry is a replacement of an inappropriate earlier repointing mortar, which may cause damage to the masonry. In this case, the original appearance of masonry and the pinnings have already been lost during the earlier conservation attempts. Mortars based on Portland cement have a strong bond with masonry and therefore are often impossible to remove without damage to the original stone. Removal of such mortar should be carefully considered. The dangers of using mortars which are too hard (usually mortars gauged with Portland cement) for repointing historic masonry have been widely publicised in conservation literature (Feilden 1998, Gibbons TAN 1 1995).

Surface finish or type of mortar pointing can influence greatly the appearance of the masonry as a whole. Feilden (1998) suggests that repointing mortar should have the same colour as the original stone and should not in any way interfere with the stone. However, the most accepted approach to the appearance of new repointing mortar is that its colour and texture should be as close as possible to the original mortar (TAN 1 Gibbons 1995, BS 6270:Part1:1982, Ashurst and Ashurst 1990). The generally accepted style of pointing of historic rubble masonry is a recessed pointing. It is preferred on the basis that it potentially causes the least damage to the adjacent masonry rather than on any appearance factor. In many cases sharp edges of ashlar masonry have been weathered and rounded, and its appearance after recessed repointing changes to square rubble like masonry (Maxwell 1994). When the mortar is recessed, it is less visible and the joints emphasise the texture of the masonry. If the masonry is pointed flush with the face of stone, the thickness of the joints significantly increases when the arrisses are worn, see *Figure 2.1*. The thickness of the joints can be also affected by the width of the pointing tools (Maxwell 2000).

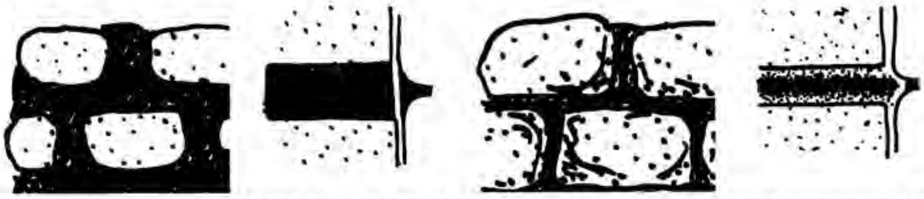
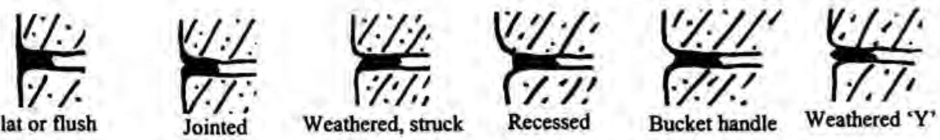


Figure 2.1: Flush and recessed mortar finish in joints, the edges of masonry are worn and flush pointing results in much wider joints.



Flat or flush | Jointed | Weathered, struck | Recessed | Bucket handle | Weathered 'Y'

Figure 2.2: Joint finishes according to BS Cleaning and surface repair (BS 6270:Part1: 1982).

For conservation purposes in general, the British Standard for Cleaning and Surface Repair of Buildings (BS 6270:Part1: 1982), specifies various types of pointing finishes for stone and brick masonry, *see Figure 2.2*. The selection of pointing finish and technique should depend on the character of masonry.

2.3.2. Rough racking

'Rough racking' is a term used for repair work carried out on areas of broken faces of masonry and wall heads. When the core of masonry and/or head of the wall are exposed to the weather elements the masonry and mortar becomes more vulnerable to water penetration. As a consequence of this, the masonry deteriorates faster since it was not constructed to be exposed.

Rough racking involves introducing new stones and re-building the exposed areas in such manner that the water can run out of the masonry without being trapped, and minimises the deterioration effect on the original masonry. The rough racking should intentionally not look like the face-work; the visual appearance of exposed mortar is much higher (Ashurst and Ashurst 1988). In general, the mortar used for rough racking is required to be durable and therefore mortar gauged with cement is commonly used in practice, especially when the mortar is used on the wall-heads (Ashurst and Ashurst 1988). Although the durability of the mortar is essential and the mortar should be to some extent waterproof, the effect of shedding water as easily as possible away from the masonry should not be underestimated. The construction and details of individual stone positions are important. The danger of accelerated decay caused by impermeable mortars is the same as for pointing. In every case, new mortar should be designed to be compatible with the existing material.

2.3.3. Tamping

Tamping is a type of deep pointing which is supposed to ensure that individual stones are firmly embedded in masonry. Joints are deeply raked out and then filled with a mortar with good bonding properties, usually Portland cement based mortar. A space is left in the joints for pointing to give the masonry a desired appearance.

For some traditional masonry this practice is completely inappropriate and it does not reflect the construction of the masonry. Such practice is based on a misinterpretation of historic masonry construction. Maxwell (2000) presented a hypothesis that rubble masonry construction could be seen as analogous to the dry stone masonry in Scotland. Dry stone masonry is structurally stable without any bond between individual stones provided by mortar. Selection of stones and their interlocking within the structure of the wall is more important for the stability of the dry masonry.

Another aspect should be carefully considered before tamping is carried out. Although the cement mortar can provide the required strength, it can damage the masonry. On the other hand, the traditional mortars (e.g. lime mortar) may not have the properties required to solve structural and stability masonry problems. For deeply deteriorated joints and decayed masonry, modern, new material and repair techniques may be the only solution. In such cases, the new mortar should fulfil the compatibility requirements of the new mortar and the historic masonry.

2.3.4. Bedding mortar, replacement of masonry units

Requirements for appearance and composition for bedding mortars are, in principle, the same as for pointing and tamping. Inappropriate and hard mortar can cause accelerated damage of original stone. The properties generally required from bedding mortar are that it should be resistant to the action of frost, rain, abrasion and chemical attack. It should be able to accommodate early settlement of the structure, have an adequate strength when hardened and good adherence to the masonry units. If the bedding mortar is not exposed there is less emphasis on its appearance and weathering quality. The bedding mortar is part of a structure and serves as a 'bearing pad' between masonry units. There has to be a sufficient bond between the mortar and masonry units and the mortar has to have a sufficient load-bearing capacity.

Design of a new bedding mortar is needed when complete rebuilding of a structure or anastylosis is considered. However, smaller scale repair works, such as replacement of individual masonry units, are more common in conservation practice.

2.3.5. Internal and external coating - plasters, renders and their repair

Plaster or render is an architectural element which may be decorative but also serves as a weathering protection of masonry. This review deals only with properties and functions of mortars related to conservation techniques where the coating serves as a protection of masonry. The fundamental properties of mortars used for coatings as a protective layer are porosity, permeability and pore size distribution. For example, porous lime renders are considered to allow moisture to evaporate and the building to breathe (Hughes 1986). Holmes and Wingate (1997) describe the same effect as the principle of a lime render, stating that the moisture will penetrate the render but will evaporate later. The durability of a coating depends on water penetration but also on the quality of materials, techniques and constructional details (Ashurst 1983).

When assessing historic masonry and its appearance in Scotland it is important to bear in mind that many medieval historic buildings were originally lime harled or rendered (Meek *et al.* 1996). The masonry was built to be coated. It therefore looked and performed differently from nowadays, when the masonry of many previously rendered buildings is left exposed. Only fine ashlar masonry was left exposed in the past. Later on, during Georgian and Victorian times, the aesthetic interpretation changed (Maxwell 1998) and exposed masonry become fashionable in architecture.

Buildings which were once rendered are sometimes considered for re-rendering. This is so particularly if the masonry has deteriorated on a large scale and individual repair of stones may not be visually acceptable. The solution can vary from just a protective sacrificial coating layer to a complete reconstruction of the façade. Render or plaster applied to protect and preserve the substrate masonry should act as a 'sacrificial surface' – it should deteriorate instead of the masonry substrate. Mortar for such a protection should be permeable to allow masonry to breathe; appropriate material is lime (Crook 1998).

Patching techniques can repair render and/or plaster itself. Its goal is a retention of maximum original material. Important factors for success of the repair are compatibility of new and existing materials including colour, texture, porosity, permeability and adhesion of the repair to the substrate.

Lime wash is another traditional technique which provided a protection to masonry. It was easy to reapply on an annual basis and it improved the appearance of façades of many farm and countryside buildings by giving them a clean and maintained appearance (Maxwell 1998). Maxwell (1998) detailed the nuances that contribute to the appearance of the lime wash, which include underlying masonry, number of coats, colour of lime putty and natural pigments. Apart from the appearance, lime wash protected the masonry acting as a weathering layer which was easy to maintain.

Most commonly the lime wash was just lime putty diluted with water but in some cases organic additives were added to 'improve' its quality. Pigments were added for different colours. Tallow was added to lime wash in Scotland tentatively to make the coating waterproof. However, an advantage of lime wash is mainly in its high water vapour permeability (Herm *et al.* 1993) which provides a balance between exterior, wall construction and interior moisture conditions. Research shows that each additive usually improves certain properties e.g. casein can act as a liquefier and increase water permeability, linseed oil affects its flow and reduces water vapour permeability (Herm, *et al.* 1993).

2.3.6. Plastic repair

Plastic repair is a repair technique which uses a mortar mix to replace and copy not only deteriorated parts of natural stones but also bricks, tiles etc. Successful application of the plastic repair technique relies on two aspects which are, however, often difficult to meet. Firstly, its appearance, which copies the original material, should, subject to weather conditions and exposure, not visually interfere with the rest of the masonry. Secondly, there has to be a good interaction and mechanical bond between the existing stone and the repair mortar, otherwise failure and deterioration of existing masonry will occur. This implies very high criteria for the mortar mix and its performance.

Ashurst and Ashurst (1988) discussed appropriate mixes for plastic repair. They pointed out that strength of mortar in itself is unimportant; more important is a resistance to wetting and drying cycles. The strength of mortar has to be balanced between the strength of the surviving stone and strength required to withstand the weather exposure. If a stronger mortar is needed, a hydraulic lime or additives or cement may be used. Mortar made of Portland cement may be needed for appearance where a higher proportion of aggregate is required. Epoxy resins and other new binders are under development. Again, all these mortars based on modern materials should comply with certain compatibility requirements before application. In any event, one of them is that the repair mortar should fail in advance of the stone.

Most plastic repair mortars are based on lime, however in some cases, such as the repair of sandstone, a negative interaction of sandstone and lime wash may occur. Maxwell (1994) described examples of deterioration based on interaction of lime and sandstone. He pointed out that in such cases pointing should be recessed and it should not extend onto the face of masonry.

2.3.7. Injection, grouting

Cavities in cores of massive walls may affect structural functions of the walls. In order to improve the strength of the masonry and bind the outer stone faces together, fine mortar is injected into the masonry core. An important part of this repair technique is a detailed analysis of the masonry and exact description of the voids.

Croci (1998) suggested that the most economic grouting is a mixture of cement, water and some fluxing and shrinkage–free additives, but at the same time he pointed out the undesirable effects experienced when the cement reacts with the original components. As a weaker but more suitable alternative to cement mixes, Croci (1998) suggested a mix based on hydraulic lime, fine sand, (pozzolan) and bentonite for fluidity. The use of pure lime mortars is limited by the length of the carbonation process in such enclosed conditions, which do not allow enough carbon dioxide to permeate into mortar. Pure Portland cement mortars are not recommended, mainly due to their high content of soluble salts and high shrinkage (Ashurst and Ashurst 1990). Other specialised mixes including lime, PFA and bentonite are often used.

2.4. Construction of stone masonry

Two basic types of historic stone masonry are generally recognised - rubble and ashlar masonry.

Rubble Masonry

Rubble masonry uses stones of various irregular sizes and shapes. Depending on the shape of the stones, the masonry could be called squared or random rubble, which can be built uncoursed or built to courses. The height of a course usually varies from 375-450mm (McAfee 1997). Massive walls were constructed as a three-leaf masonry.

Ashlar Masonry

Ashlar masonry is made of rectangular stone blocks of the same height (in one course). Current ashlar masonry is precisely cut with very fine joints, no more than 4.5mm (Hill 1995). Ashlar masonry can also have one or two faces with mortar and stone rubble infill between them.

Roman Masonry

Across Europe a large number of historic structures were built as a three-leaf masonry (Toumbakari *et al.* 2000). This developed from Roman stone masonry, which was constructed as multiple-leaf masonry, i.e. two faces of stone curtains and the core between them filled with mortar and stones of various sizes. Originating from around the 3rd Century BC, this technique is known as 'opus caementicium', 'opus reticulatum' or others depending on the shape of the stones used (Samuelli Ferreti 2000). The core mortar between stones often possessed some hydraulic property. Core mortars from Byzantine masonry are known to contain pozzolanic additives such as brick dust and crushed bricks (Binda *et al.* 2000).

Toumbakari *et al.* (2000) studied a three-leaf masonry on a model wall. The authors reported that the compressive strength of the wall is affected by the quality of the mortar and the filling materials, not by the compressive strength of stones or bricks. The authors confirmed that low strength mortars were often used as the masonry core infill. However, the poor quality core mortar did not seem to lower significantly the overall strength of masonry, as would be the case for modern single-leaf masonry structures.

Other studies (Maxwell 2000) showed that as the stone walls were usually of a considerable thickness their stability depended on the construction technique and selection of stones. Maxwell (2000) suggested that in Scotland the traditional method of masonry building derived from dry stone masonry. In lime and stone walls the mortar helps to spread the vertical load but acts as a seal between stones to protect the masonry and whole building against weather conditions (e.g. ingress of water). However, analogous to the dry stone walls it is the selection of stones, binding and their fitting together which provided the stability of these structures (Walker 1993).

3 COMPATIBILITY

The reality of masonry conservation practice is that stone or brick is treated and preserved while the mortar (pointing, bedding) is replaced or partly replaced and therefore lost. Although the value of original mortar is recognised at present and the mortar is preserved whenever possible, it is often necessary to repoint or apply another repair method utilising a new mortar mix in order to prevent a further damage of the masonry units. Any new material and treatment introduced to a historic fabric should, however, be compatible with the fabric. Compatibility is relatively easy to understand in general, but its interpretation to a real conservation task may be rather indeterminate. Compatibility can be comprehended from several points of view. It should be in agreement with the philosophical and ethical issues in conservation. Depending on the actual design of mortar it can be regarded as traditional or scientific. These aspects are sometimes thought of as distinct, but in fact they are highly interrelated and complementary.

3.1. Definition of compatibility

Bell (1997) in the 'Guide to International Conservation Charters' published by Historic Scotland (TAN 8) explains the term 'compatible use' based on the definition of the Burra Charter as:

'a use which involves no change to the culturally significant fabric, changes which are substantially reversible, or changes which require a minimal impact'.

In a simple description of compatibility, Teutonico *et al.* (1997) state that the 'introduced treatments or materials will not have negative consequences'.

Van Hees (2000) suggested a definition of compatibility related directly to mortars as follows:

'The new mortar should be as durable as possible, without (directly or indirectly) causing damage to the original material'.

The mutual subject of these three definitions is that any material can be used as far as there is no further damage to the original material. Technically it means a consideration of mechanical, physical and chemical properties of new and original materials and their interaction. On the other hand, strictly speaking, a new mortar which would not cause any damage to the original masonry may still not be compatible. Colour, texture and aesthetic values should be considered and usually the selection of a new repair mortar is based on the 'like for like' philosophy. However, any modern or 'dissimilar materials should not be ruled out' as long as there are 'no negative consequences' (Teutonico *et al.* 1997). The Burra Charter deals with this problem by a general limitation of 'no change to the cultural significance'. 'Minimum impact' or 'as little as possible' are general rules of repair and conservation philosophy that should be maintained for any conservation treatment.

There are still mortars currently applied in remedial conservation works which can cause damage or accelerate deterioration to the historic substrate. Therefore the primary aim of these definitions is to be able to determine materials which are appropriate ('at least' not to cause any damage) for use in conservation by considering many different aspects. This leads to a technical understanding of compatibility as specifications or general requirements for design of a new mortar based on material characteristics.

3.2. Compatibility with regard to conservation philosophical and ethical issues

Basic conservation guidelines are formalised by international conservation charters and standards. The problem of compatibility was perhaps officially introduced by the Athens Charter (1931) which approved the use of modern materials for restoration of monuments. Later on, the Venice Charter (1964) appended that modern techniques for conservation can be used if supported by experience and scientific data, and where traditional techniques were proved inadequate. The Venice Charter stressed that restoration should respect the original material. It also stated that any replacement should be distinguishable from the original so that restoration does not falsify the artistic or historic evidence. This requirement may be in contradiction with the most widespread current approach of designing a compatible mortar as a copy of the original one. Another important point was made in the Declaration of San Antonio (1996) about the authenticity of materials. It expressed an idea that only the historic fabric is authentic and the restored fabric is not. However, it acknowledged that some materials (it was not specified, but lime mortar could be considered one of them) do weather and require a periodic replacement or maintenance. In such cases, the traditional techniques should be followed.

Philosophical and ethical issues in conservation are very complex and develop with society. They apply as guidelines in general but they should also be able to provide a specific answer about policy and conservation treatment for every individual structure. Every historic structure is unique and so are the values to be preserved (Burman 1997). The values to be preserved primarily define and limit functions and properties required from the conservation treatment. In the case of this review they limit and define functions and properties of repair mortar. The cultural significance of the structure enshrines all its possible values (emotional, cultural, architectural etc.) and its formulation leads to a determination of correct conservation policy (Burman 1997). For example, a conservation treatment of a fresco painting lies in the preservation of the artistic value of the work rather than the authenticity of the mortar. However, such a conservation approach is not an inflexible rule - it should be established individually through the cultural significance of the work. This example may be seen as an obvious extreme, but it also illustrates the point that the authenticity of historic mortar is often 'sacrificed' to preserve some other values. The most common situation of where historic pointing or bedding mortar is "sacrificed" is in connection with structural stability and the integrity of masonry; and hence the preservation of the whole structure. Another example is the weathering of pointing mortar, which is supposed to protect the masonry as a form of seal. Therefore, its replacement when even partially damaged is a logical step of any building maintenance.

However, when a new mortar is used to repair original masonry it must not damage the host mortar and it should attract all the adverse agents before they affect the original material. However, the colour of a new mortar could be a matter of whether it should or should not be visually distinguishable from the original mortar, especially when a 'like for like' approach is applied. Also, when a different, modern material is used for repair, should it try to match the original colour and texture? The Athens Charter suggests concealing consolidation whenever it is possible 'in

Figure 3.1: Tantallon Castle and detail of its deteriorated masonry. There are many historic buildings with repointed masonry, which have undergone some preservation treatment that resulted in a loss of original pointing and bedding mortar. For example Tantallon Castle with its 4m thick, red sandstone ashlar curtain walls which were repointed and consolidated before the Second World War. The appropriateness of this kind of intervention may be questioned. However, the work was done with the best knowledge and skills of that time. On the other hand, how much of the original mortar was there when it was treated? Pointing mortar is often the first to deteriorate so repointing has been a common practice in building maintenance in the past. The compatibility of such treatments and application of traditional techniques should be considered.

order that the aspect and character of the restored monument may be preserved'. Although this part of the Athens Charter may now be seen as a bit obsolete, as consolidation work is clearly visible on most historic structures, the character of the repointed masonry is certainly a part of its value and should be preserved.

Figure 3.2: Sandstone masonry has been repointed with cement gauged mortar where a technique called 'Stirling grit wash' was applied to make the repointing look more weathered by exposing the aggregate. Although the pointing is done to resemble historic, weathered surface masonry, repointing in this or some other modern technique can be easily distinguished by any careful observer. However, when a traditional technique and materials are used, the distinction after some time and when it weathers, is much more difficult to make, without more detailed analytical techniques. Tantallon Castle, pound coin for scale.

The views on each individual conservation task vary according to national conservation policies. In Scotland, Historic Scotland published 'The Historic Scotland Guide to International Conservation Charters' (TAN 8, Bell 1997). It explains and discusses the basic conservation terms in a context of international conservation charters. For practical conservation tasks, reference should be made to 'The Repair of Historic Buildings in Scotland' by Knight (1995) where the principles of repair adapted by Historic Scotland and reflecting Scottish practice can be found.

Compatibility is now becoming an increasingly important issue. During the Dahlem workshop 'Saving our Architectural Heritage: The Conservation of Historic Stone Structures', Sass and Snethlage (1997) pointed out that today strict reversibility must be replaced by compatibility and 'retreatability' measures. If that should become standard practice then there is a need for compatibility to be better understood and defined. Compatibility should describe the properties which newly added material or original material after conservation treatment should have in relation to the original material. Research into compatibility of mortars is still in its infancy and no guide or standardised techniques for determination of the correct material for repair are available.

The current approach towards the design of a compatible mortar can be found in practical conservation literature. It offers general requirements for the formulation of restoration mortars based on generally accepted conservation principles, basic physical properties and compositional requirements for new mortars (e.g. Maxwell 1998 and Ashurst and Ashurst 1990, Ontario Ministry of Citizenship 1988, Feilden 1998, Croci 1998). The principles are derived from the general consideration of the properties of masonry materials. However, it is rare to find specific technical rationales behind conservation practitioner driven requirements, and justification with reference to published research is also rare. This may be because such research is itself rare (Válek 2000).

Groot *et al.* (2000) divided the approach to specify new mortars into 'traditional' and 'modern'. The 'traditional' approach is based on the use of traditional material which is subsequently altered or treated in order to achieve all compatibility requirements. On the other side, there is the 'modern' approach based on compatible requirements to develop a formulation of a new mortar, and if necessary it would use modern materials. Groot *et al.* (2000) concluded that mutual understanding of both approaches would lead to better development of mortar for conservation.

3.3. Compatibility with regard to traditional approach and conservation practice

Practical experience gained from conservation projects is a valuable source of knowledge about performance and behaviour of mortars. 'Hands on' experience is highly valued in conservation and practical experience is considered to be particularly useful for the design of a compatible mortar.

A review of contemporary practice of using lime mortars in conservation was presented at Historic Scotland's International Lime Conference (Ward & Maxwell 1995). Lime-based mortars in particular are good examples of a traditional material used in conservation, where good skills and practical experience have a significant influence on performance. Burman (1995) described the advantages of using lime in various conservation techniques and the successful revival of lime into wider conservation practice. For traditional lime-based mortars, often the conservation technique, good site practice, workmanship and skills are considered to be the key instruments for their successful application (Gibbons 1995, Johnston 1995). Many failures of lime-based mortars are caused by their inappropriate use or by a lack of practice and training in their correct application (Gibbons 1995, Holmström Part 1, 1995).

The most basic specifications for new mortars are from practical building and masonry conservation guides (e.g. McAfee 1997). The usual advice is to carry out basic analysis to obtain the composition of the mortar (usually by dissolving the mortar in an acid) in the first instance and, if the original mortar has performed well, then the new mortar mix should resemble it as closely as possible. However, it is advised that the new mortar should be softer than the stone. Ashurst (1990) in the 'English Heritage Technical Handbook' specifies that the original aggregate should be copied, however, the mix may need to be modified to improve the weathering characteristics. Milner (1972) points out that natural materials should be used to match the colour and texture. He also suggested that soft mortars should not be used for repairs on buildings originally constructed with hard mortar.

The basic specifications for mortar required by conservation practice can be summarised as follows:

- The formula of the new mortar should match that of the original one; natural materials should be used when matching colours and textures.
- The new mortar should be softer than the original mortar or masonry but, on the other hand, the mortar should not be too soft if the original masonry was constructed with hard mortar.
- Under no circumstances should the new mortar cause deterioration to the existing host material (mortar, masonry).
- Side-effects and long-term effects caused by repair should also be considered. For example, new plastering or repointing may change the indoor moisture conditions. Larsen (2000) described the importance of stable moisture conditions inside a chapel with salt-contaminated painted plasters in Denmark.

These basic requirements are applicable in general and can be extended depending on the particular function and application of the mortar. For instance, Holmström (1992) presented a list of compatibility criteria for lime mortar renders used by contractors in Sweden.

- Materials and components used must be removable, and must not change the physical or chemical balance of the building and must not change the aesthetics.
- Each layer of render (mortar) should be weaker than the substrate.
- Materials with the same properties as the original should be used. If the original materials are not used, this must be justified and all relevant properties of the original and the substitute must be declared.

These suggestions enshrine minimum intervention and the preservation of existing historic fabric. In addition, Holmström's (1992) criteria emphasise the need for reversibility of the material applied. The final point arrives at the heart of the compatibility issue and the desire of conservationists to replace like with like. It makes demands for analysis of historic mortars in order to formulate compatible replacements that resemble the original. Basic performance testing and in-situ trials are common and recommended. However, there is less demand for testing and characterising the new mortar properties, as it is a copy of the original one and therefore it is assumed to perform in the same way. Such assumption may not always be correct. Válek (2000) in his PhD thesis discussed a theoretical compatibility model for two mortars, one designed on a 'like to like' basis and one on a 'compatible properties' basis. The performance of both mortars relied strongly on the curing and ageing conditions. However, the curing conditions of the new mortar may not match the ones of the past.

3.4. Compatibility with regard to physical, chemical and mechanical properties

Alongside the traditional approach to the specification of a new mortar there is a growing need for the scientific description of its compatibility, as was concluded by the ICCROM conference in 1981. It appears that the selection criteria for new mortars should be based strictly on material characteristics of the mortars, providing that the mortar complies with the conservation requirements and principles. The need for exact description and characterisation of the mortar led to a more technical description of compatibility. It was reflected in some specifications for new mortars, as they became more detailed and technical. For example, Historic Scotland's Technical Advice Note 1, Preparation and Use of Lime Mortar (Gibbons 1993) defines that the new mortar should have porosity and strength close to the original one. It should be less dense and more permeable than the host masonry and at the same time it should be sufficiently durable.

When a new compatible mortar is to be designed the required properties must first be determined and consequently confirmed by testing. The technical design of new mortar can be divided into two main activities.

- Analysis of the original mortar

Analysis should provide all the information about the historic mortar (and masonry) that is needed for the design of a new compatible mortar. In current practice analysis most commonly consists of only the composition of the historic mortar. However, recent research suggested that analysis is more complicated than it was thought to be. Hughes *et al.* (1998) pointed out that the complexity of historic materials that reflects their production, preparation and weathering over the long term may mitigate against the recognition of the properties of the original mortars at the time of their application. Therefore, only the 'contemporary' properties and composition of the historic mortars are known and simply copying these historic mortars without the knowledge of their change may not lead to the same material performance quality. It is important to note that analysis can help to identify other important influential factors, for example mixing (Leslie 2000). There should be complex analysis of the whole masonry, building, etc. in order to provide all relevant information.

- Specifications for new mortar, its properties and testing

The specification of a mortar from the point of view of technical compatibility is based on comparison of properties. Preliminary research into this field was carried out by Peroni *et al.* (1981) and presented during the ICCROM symposium in Rome in 1981. From the beginning, the authors pointed out that it was not possible to set down a list of detailed requirements for ideal conservation mortar, but instead general principles were used. The authors proposed a number of tests (flexural strength, compressive strength, modulus under compression, alkaline elements concentration, total porosity and pore size distribution) which were carried out to test specimens made of various mortar mixes including lime putty mortar, hydraulic lime mortar and mortars gauged by cement, brick dust and pozzolanas. Although the paper neither came to any conclusion about what properties were important nor determined the tests for selection of a new mortar, it set up a baseline for further research.

Sasse and Snethlage (1997) studied methods for the evaluation of stone conservation treatments. They identified properties to evaluate compatibility of repair mortars and stone masonry. They also introduced preliminary tolerance limits established for various materials and property tests.

Stone Repair Materials

Property	Symbol	Requirement (after 1 year)
Dynamic *E*-modulus	*E*-modulus	20-100% (60)
Compressive strength	β_{CS}	20-100% (60)
Thermal dilatation coefficient	α_{TH}	50-150% (100)
Water uptake coefficient	*W*	50-100%
Value of water vapour resistance	μ	50-100%
Pull-off strength	β_{POS}	0.5-0.8% β_{POS} stone

Table 3.1: Investigation method and requirements (as a percentage of the value of the substrate) to evaluate stone repair materials. The requirements are related to the properties of the substrate (From Sasse and Snethlage 1997).

3.5. Compatibility with regard to mortar and masonry interaction

Interaction between mortar and masonry is an effect between them which results in some physical or chemical change to one or both of them. It is as an evaluation of compatibility performance. Bad interaction means that incompatible materials or repair techniques were applied. It results in deterioration and accelerated weathering of masonry material. However, the use of an incompatible mortar or treatment does not mean bad interaction a *priori*, it merely brings about conditions favourable for deterioration. The deterioration itself happens through environmental agents. For example, a dense cement mortar containing salts used for repointing of porous sandstone masonry may not cause any bad interaction without the presence of moisture in very dry ageing conditions. Water or moisture is needed for a chemical and most physical deterioration processes to take place (Collepardi 1990). Summarised by Amoroso & Fassina (1983), water is the main cause of degradation mechanisms for masonry materials.

When testing a new compatible mortar the interaction with masonry should be considered in a wider context comprising all the potentially influential factors. It should be kept in mind that properties of mortars when determined on small specimens in the laboratory are different from the real masonry conditions in-situ (Depraetere *et al.* 2000). Therefore, full-scale 'in-situ' trials are important for real evaluation of the interaction. Full scale testing should be preferred in any larger conservation tasks (Stewart *et al.* 2001).

Figure 3.3: Small trial panels for mortar and masonry built on site help to assess the appearance of a replacement mortar and also its longer term durability, and compatibility with the associated materials.

Top: Small test wall panel at Tantallon Castle, that was also intended to test replacement stone.

Bottom: Trial panel of repointing at Inverlochy Castle.

On the other hand, it should be emphasised that degradation connected to interaction of mortars and masonry is not the sole problem of new, modern replacement mortars. Maxwell (1994) described cases of deterioration related to the interaction of original lime mortars with sandstone and/or granite. Soiling and decay in the vicinity of joints observed on masonry of many structures suggest the interaction of the lime mortar and stone. Maxwell (1994) described cases of ashlar masonry being changed to square rubble-like masonry due to decay round the joints. The deterioration was caused by the conditions in which the masonry had been ageing.

Research into properties related to interaction of materials is driven mainly by the deterioration of the materials (see *softer mortars).*

A number of cases of masonry deterioration could be put down to salt formation and crystallisation. Rainwater can transport soluble salts from mortar to adjacent stone (Perry & Duffy 1997). The interface zone between two different materials is the place where potential salt crystallisation can occur (Baronio and Binda 1987). The exact position of the salt crystallisation depends on capillary migration between mortar and stone, and rate of evaporation (Charola & Lazzarini 1986). If the mortar is more porous than the stone, evaporation and salt crystallisation take place mainly within the mortar and vice versa (Charola & Lazzarini 1986).

Rainwater, 'acid rains' can facilitate the formation of gypsum and other salts (Charola & Lazzarini 1986). The susceptibility of mortars to react with air-borne sulphur dioxide was measured in a weathering chamber by Zappia *et al.* (1994). The authors concluded that the reactivity is related to porosity, specific surface and alkalinity of mortars. Perhaps surprisingly, the reactivity did not correlate to the quantity of calcium carbonate. The results suggested that the most reactive was a cement based mortar (lowest quantity of the Calcium Carbonate) followed by lime-based mortar, and the least reactive was a lime-pozzolana mix (Zappia *et al.* 1994).

In general it concentrates on the use of materials and their compatibility. However, a detailed understanding of degradation requires study of its mechanisms. Particular degradation mechanisms are summarised below, but for masonry the water/moisture transport is the most crucial to understand. The way water transports through an interface between masonry units and mortar significantly affects the moisture behaviour of the whole historic masonry. For example, Depraetere *et al.* (2000) examined the influence of the mortar/brick interface on moisture transport. The authors described a zone formed at the interface with different porous structure and related moisture properties. They also pointed out that mortar cured in a joint between bricks has other moisture properties than the mortar cured in moulds. This indeed strengthens the argument for in-situ trials when assessing the compatibility and interaction of different materials.

Another mechanism of masonry degradation is caused by different expansions of masonry materials. A case study on defects of renders of a cathedral in Toledo, Spain (Macías 1992), showed that the differences between thermal and "hygric" (sic.), or *hygroscopic*, expansion of gypsum, lime mortars and dolomite stone caused cracks, fissures and spalling off of the render. (Hygroscopic refers to the tendency for a substance to take up moisture.) Lime mortar was found to have low relative thermal and hygroscopic expansion (Macías 1992). Vermeltfoort *et al.* (2000), who studied thermal expansion of mortar repointing and its compatibility with original masonry, described that the interaction between brick, bedding mortar and two types of repointing mortar causes irregularities in the stress and strain distribution which consequently has an effect on the durability of the mortar and masonry. They concluded that strong pointing mortar expands more than soft bedding mortar. This represents one of the few efforts to quantify compatibility and demonstrates conclusively that stronger, harder cement mortars are less appropriate, in one physical aspect, for the repair of historic masonry than softer mortars.

A way of studying building materials from a conservation science point of view is to study their damage mechanisms and their deterioration (e.g. Torraca in Porous Building Materials, 1988). Possibly the best model for scientific studies of deterioration of masonry materials is based on a review of literature on deterioration of porous materials published by ICCROM in 1976 (Stambolov and van Asperen de Bore 1976). Torraca (1988) later on in 'Porous Building Materials' described material deterioration under the following categories:

- External mechanical deterioration. This is caused by excess of stress with respect to the strength of the material (load, thermal expansion, stress caused by transport or working techniques, dynamic load and vibration). When excessive stress occurs, the material cracks and even small hair-cracks can lead, in combination with other deterioration factors, to accelerated deterioration.

- Internal mechanical deterioration. This is sometimes called physical deterioration and is mostly due to a physical variation of water inside masonry like evaporation, capillary flow. A large stress can arise inside the pore structure when water freezes and crystals of ice or minerals are formed within the originally water filled pores damage caused by salt crystallisation and efflorescence or similar effects.

- Chemical deterioration. This is mostly connected with a reaction between sulphate and the other compounds in the masonry (Collepardi 1990) Chemical corrosion almost always requires the presence of water (Torraca 1988). Water can play two roles in chemical corrosion:

  (a) water in the form of liquid and vapour is chemically active

  (b) water in the form of liquid acts as a transport medium for other components

  Water which has been in contact with other solid material of the same kind is not chemically active (rising damp) but it can still act as a transport medium for other deterioration agents, e.g. see physical deterioration. The danger of chemical corrosion increases with atmospheric pollution and acid rains (Charola 1986).

- Biodeterioration. This can be caused by bacteria, algae and/or fungi which produce acid. Lichens can also penetrate into several millimetres of the surface of the material. Moss commonly grows on the surface of alkaline materials (lime mortar, Torraca 1988). Roots of higher vegetation can penetrate deeply into joints and cause deterioration of the masonry.

Table 3.2: Deterioration of masonry materials

4 ANALYSIS OF HISTORIC MORTARS

The analysis of historic mortars is carried out broadly for two reasons (Hughes & Callebaut 2000):

- For specific conservation and repair related investigations, looking to select replacement materials and/or determine the cause of evident problems in the deterioration of a historic building to allow the formulation of conservation and repair strategies.
- Academic studies looking to clarify the architectural, chemical and physical performance of historic mortars for the development of replacement materials or the archaeological study of building technology and its associated social implications.

The objective of testing for characterisation may primarily involve the determination of the essential properties of an old mortar, with a view to the formulation of the composition of an appropriate repair mortar. It may also be aimed at the exploration of the potential and limitations of a testing technique used for characterisation (Groot *et al.* 2000). For example this can involve (Groot op. cit. 2000):

- The identification of the limitations of testing methods, including wet chemical analysis (Bläuer Böhm 2000; Martinet *et al.* 2000) and differential thermal analysis (papers by Moropoulou *et al.*, Ellis 2000).
- The problems of the identification of types of additives and natural hydraulic lime components in old mortars (Sickels 1987, Krist *et al.* 1999, Van Balen *et al.* 2000; Charola 2000; Callebaut *et al.* 2000) and their differentiation.
- The identification of the provenance of natural hydraulic lime (Callebaut *et al.* 2000).
- Diagnosis of the cause(s) of damage focused on a better understanding of damage mechanisms (van Hees 2000; Blanco-Varela *et al*; Larsen 1999).

The following sections deal first with sampling of mortars, and then with the variety of techniques currently used for the characterisation of the properties of historic mortars, both compositional and mechanical.

4.1. Sampling, damage diagnosis, hypothesis formation and on-site visual analysis

The first stage in the analysis of mortar materials is a careful pre-analysis of the conservation objectives of a study or conservation effort. This is particularly important as historic buildings are special cases. Broadly applied renovation or wholesale replacement is not appropriate, and as the historic fabric is the valued historic and social material it must be protected as much as is practicable (Hoffman 1998). This places considerable constraints on the analyst and the sampling operative (Hughes & Callebaut 2000, op. cit.).

Two major controls operate on sampling practice: the objectives of an investigation and the analyses (physical, chemical or descriptive) needed to fulfil the objectives (Hughes & Callebaut 2000). The objectives of a study must be clearly stated before any sampling can take place. Minimum action is required in order to reduce effort and cost and minimise destructive intervention and damage. The materials, the construction method and stratigraphy of the building must be understood thoroughly before sampling begins, in order to ensure the correct materials are sampled and so that sufficient material can be obtained. Spatial and temporal distribution of materials must be understood to do adequate sampling (Binda and Baronio 1989, 1991, Pursche 2000, Hughes & Callebaut 2000, Blauer Böhm 2000, Leslie and Gibbons 2000, Cardoso 2000, Sass 2000).

The formulation of hypotheses regarding damage mechanisms (van Hees 2000, op. cit.), the decay state of the building (Long *et al.* 1998) and the subject of study directs the choice of analytical method which in turn has a requirement for a minimum quantity of sample, but also a certain quality of sample, whether as a coherent lump preserving textural-component relationships or as a powdered sample. The choice of analytical method determines the sampling requirements (Hughes & Callebaut; Goins; Andersen; all 2000).

Visual and other non-destructive evaluations assist in the assessment of ideas about the variations in the macroscopic characteristics of mortar materials, allowing choices to be made on the representative nature of sample sets (Cardoso 2000, Válek *et al.* 2000, Long *et al.* 1998). It is accepted that for historic structures the opportunities for statistically adequate sampling are limited due to the culturally precious nature of the materials. Samples should be representative, but if this is not possible the degree of bias must be understood (Hughes & Callebaut; Goins, Sass; all 2000). Sampling should be done by experienced people, who know what is required to achieve a specific test objective, and they should preferably have experience with the experimental technique and its sampling demands.

British Standard BS 4551: Part 2: 1998 "Methods of testing mortars, screeds and plasters" sets guidelines for the sampling of hardened mortars. This standard is aimed primarily at the characterisation of mortar in more modern buildings, but the approach is applicable to older historic buildings. The main reasons considered for sampling are the variability in different parts of the work, the composition at specific, often problematic points, and the average composition over an area of masonry. Where an averaged sample is needed to represent a large area or the sample needs to reflect variation, a large sample set is required. Minimum sample sizes are specified as being representative per $10m^2$ for brick/block work, plastering and floor screeds, and the method of obtaining a combined bulk sample is given. When sampling masonry mortar the whole thickness through the wall should be sampled, requiring the removal of masonry units, something not always feasible with a historic building. Where it is not possible to remove bricks or blocks to take a mortar sample, drilling can be used to generate a powder which can be used for a chemical composition, though this can cause segregation of materials and result in a non-representative sample.

Ashurst 1998 states that the samples taken "should be the minimum necessary to gain the required information without doing damage to the historic structure". Technically of course taking any sample is doing damage, but this needs to be considered against the benefits gained by sampling and gaining additional information through analysis. Ashurst goes on to specify that sampling must be performed by someone familiar with the building, and that the analyst should also be involved. The sample itself should be of at least 50g and in a coherent lump. The location of the sample must be precisely recorded and sufficient sample must be taken in order to ensure some form of representative analysis. Ashurst also gives an example of a data-recording sheet to be completed whilst sampling. A similar form is also given by Hughes and Callebaut (2000).

Ashurst (1998) and Hughes and Callebaut (2000), emphasise the usefulness of first performing an on-site visual analysis of mortars, before physical sampling. A good idea of the general components of the mortar can be derived by an experienced person using a low level of optical magnification. Binder, aggregate and other inclusions, including particular mortar additives, can be recorded in this way. Ashurst goes on to suggest gentle scraping to remove weathered surfaces to improve identification of components. This analysis will also clarify the method of construction and the profile of the joint, as well as highlighting any repointing or other later repairs. Groot *et al.* (2000) suggest that features such as the number and thickness of coats or applications of bedding mortar or renders, the presence of cavities, fractures, cracks and macro porosity within the binder, the presence of hair and other additives and the abundance and distribution of unmixed binder (lime lumps) can be observed. Also the lithological characteristics of aggregate and pozzolanic additives. The general type of binder (hydraulic, cement, lime etc.) and the nature and size of the aggregate will be identified.

4.2. Methods used for technical characterisation

A number of techniques used for the characterisation of old mortars are presented below with the basic properties which can be determined by the methods, and their limitations. For each a selection of papers is reviewed. It must be borne in mind however that the separation of these techniques does not suggest that they are applied in isolation. Many of the papers reviewed use several methods of analysis to characterise mortars, as often the identification of a component or property is not unequivocal using one method alone.

4.2.1. Acid dissolution and wet chemical analysis

The simplest form of mortar analysis involves the separation of the aggregate from the binder to determine their relative proportions to allow a replacement mortar to be formulated. This form of analysis also allows the qualitative determination of the type of binder and the characteristics of the aggregate or other additives. The carbonate binder can be dissolved from the aggregate by the use of a dilute acid, most commonly Hydrochloric Acid (HCl). The main limitation of this technique is that if carbonate aggregate is present in a mortar then it will be dissolved along with the binder (Ashurst 1998, Leslie and Gibbons 2000). In Scotland almost all aggregate is composed of silicates which do not dissolve readily in acid, thus permitting its use for the majority of mortar characterisations. Ashurst (1998) gives a simple procedure that can be followed. It is often possible to get a good, though not exact, idea of the degree of hydraulicity of a mortar, the presence of cement and the use of admixtures and pozzolanas using acid dissolution combined with careful visual analysis before and after.

The use of dissolution analysis also leads on to the further application of standard chemical analysis techniques. As the binder is put into solution this makes it amenable to instrumental chemical analysis. The soluble silica content can be determined, which is related to the hydraulicity of the mortar (van Balen *et al.* 2000). Soluble silica and other element concentrations can be analysed by various methods including, AAS, ICP, ion chromatography. However, the presence of pozzolanas in mortars may disrupt the determination of the original composition by wet chemical analysis, by changing the distribution of soluble silica due to soluble reaction rim characteristics around pozzolana grains (van Balen 2000, Charola 2000). Pozzolanas will also be reduced in size through acid digestion because of reacted rim compositions, resulting in inaccurate grain size distribution determinations (Bläuer Böhm 2000).

However, acid dissolution has not been standardised and there is some debate over the exact method that should be followed. This is particularly important for full elemental chemical analysis where the method of sample dissolution has been shown to alter the concentrations of elements in samples. Van Balen *et al.* (2000) demonstrate that the measured soluble silica varies with the temperature and the strength of the acid used in dissolution, due to contributions from aggregate and other additives. Stronger acid and higher temperatures of attack resulted in higher measured SiO_2 contents and relative errors up to 100%. The mineralogy of the aggregate was also proven to have an effect, micas and feldspars being more susceptible to solution by acids than pure quartz.

Alvarez *et al.* (1999) recently looked at the effects of hot HCl attack on historic mortar analysis. The same acid concentration was used to analyse the same samples, but in one test at room temperature with mechanical stirring, and in the other hot acid was used also with mechanical stirring. Some significant differences in result were obtained. The hot acid method was found to dissolve a greater portion of the binder, as revealed by CaO contents of the soluble fraction and the total percentage of the insoluble residue. For the hot acid attack the insoluble residue contained no remnant CaO. Significant reductions in total Fe, Al and Ti oxides were also seen in the insoluble residue from the hot acid method. The conclusion is that for quantitative analysis using acid dissolution, the method must be chosen carefully and clearly stated. More significantly, however, the recorded amounts of insoluble residue, most often used as a measure for mortar formulation on a qualitative basis, also showed significant differences dependant on the method applied.

4.2.2. Optical microscopy/ Petrographic analysis

The use of optical microscopy is a powerful technique for the investigation of the components of historic mortars. After sampling, mortar is cut, mounted onto a glass slide and ground to a thickness of 30 microns (30/1000 of a millimetre), forming a "thin-section" that permits the transmission of light through the components of the mortar. This is best performed on coherent samples of mortar that preserve the full texture of the original material, though powders are still amenable to compositional investigation using this method if mounted in a resin before cutting and grinding. Samples are commonly impregnated with a coloured epoxy resin to ensure integrity during cutting. The thin-section can be examined using a petrographic, polarising, microscope. A specialist investigator familiar with mineral recognition can then document the composition of the aggregate, binder (including anhydrous clinker), additives (organic and inorganic including pozzolans) and secondary mineral formation including salts. The two-dimensional cross-sectional texture of a mortar can also be described, for example the size and shape of the aggregate and porosity. This form of analysis is called "petrographic" analysis.

The study of hydraulic cements using petrographic techniques, both of fresh clinker and hydrated cement pastes in modern mortars and concretes is well established. Campbell (1986) provides a comprehensive guide to the recognition of hydraulic clinker phases in reflected and transmitted light. St. John *et al.* (1998) also cover the subject of concrete petrography very thoroughly, in what is often considered the standard work on this subject. Petrographic studies of concrete often focus upon aspects of durability (e.g. Idorn 1967). These include sulphate resistance (e.g. Hooton 2001, Hagelia *et al.* 2001, Sibbick and Grammond, 2001, Oberholster *et al.* 1984) and issues such as determining the water:cement ratio (Elsen, 1995, St. John *et al.* 1998).

The petrographic examination of historic mortars and concretes has lagged behind that for concrete, but has risen in importance over the past few decades. Idorn and Thaulow examined a very early Portland Cement concrete from England dated 1847, made from Portland Cement produced by William Aspdin. They were able to identify through microscopy the nature of the aggregates, but also that the cement produced was coarsely ground, with a low water:cement ratio, deliberately air entrained and well compacted, resulting in a highly durable material. The concrete was only carbonated to a depth of 5mm. Unhydrated and hydrated fragments of clinker were also observed preserved in the cement phase.

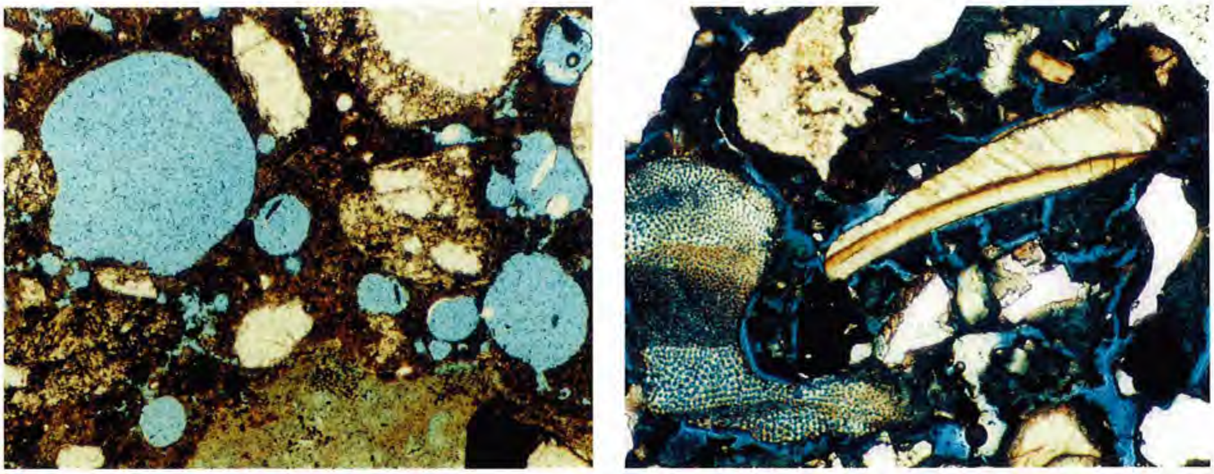


Figure 4.1: Two photomicrographs of old (16th C left, from Lochranza Castle, Arran) and new (approx 1995, right, from Inverlochy Castle, Highlands) mortars. Both images were taken in Plane Polarised Light and show how the distribution of binder (B), aggregate (A), porosity (P, in blue) and other components within a mortar can be imaged using a polarising petrographic microscope. The old mortar shows distinctive large rounded pores and the edge of a lime inclusion to the bottom of image. The new mortar shows a more homogeneous binder phase, thin, arcuate shrinkage cracking and in this case two fragments of carbonate shell material (echinoid fragment, left, and mollusc shell fragment, right). The field of view in both images is about 4mm. This form of analysis allows for determination of binder to aggregate ratio using point counting or image analysis.

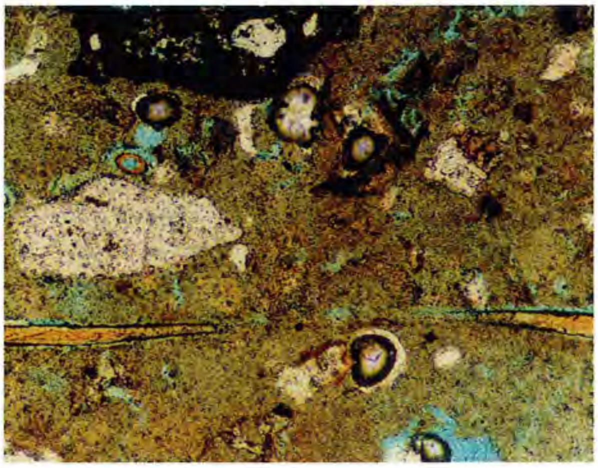


Figure 4.2: Photomicrograph of internal plaster from early 18th Century. Section through hair in plaster is clearly visible, running lower centre from left to right across image. High binder content is apparent. Field of view 1mm.

Rayment and Pettifer (1987) studied lime mortar taken from the core of Hadrian's Wall using a variety of instrumental techniques. Using optical petrography they were able to describe the aggregates, the rock types of the masonry blocks, and the presence of hydraulic C-S-H phases. In a paper on the petrography of some ancient Indian plasters, Karanth *et al.* (1986), describe the types of aggregates present, commenting that they were apparently screened for size before use, and the presence of pieces of unburnt carbonate interpreted as the raw materials used for preparation of the lime.

The study of ancient mortars has been stimulated in part by the search for stable, highly durable materials in which to store nuclear wastes. Langton and Roy (1984, and reports referenced therein 1983) analysed ancient Roman and Greek mortars, using optical petrography as part of a wider analytical scheme. They were able to identify the aggregates and other, mostly pozzolanic additives and the degree of carbonation of the material. Rassineux *et al.* (1989) also studied Ancient Roman mortars from this perspective, combining optical petrography with Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD). Optical studies confirmed the degree of carbonation and the location of secondary degradation products such as ettringite.

Middendorf (1991) presents thin-section analysis of historic mortars in the context of a comprehensive analysis scheme involving chemical analysis and XRD. He compares the petrographic appearance of modern lime, gypsum mortars and Portland Cements with historic equivalents. Aggregates including shells, wood, charcoal, anhydrite, brick and gypsum are identified by petrographic analysis. Baronio and Binda (1987) also use optical methods to study the interface between binder and aggregate and "cocciopesto" (brick dust pozzolan) additives. The brick dust and flint aggregate were observed to have reacted with the lime to form a reaction layer surrounding the grain. Cement-based mortars were observed visually to have a poor bond between brick dust and the binder, with large voids present. Lime mortars displayed a much better bond between the brick dust and the lime binder.

Optical petrography has also been used to identify hydraulic components in 19th Century historic mortars from Leuven, Belgium, by Callebaut *et al.* (2000), backed up by XRD and Scanning Electron Microscopy.

The presence of gehlenite (C_2AS), alite (C_3S) and belite (C_2S) indicate that the material analysed was a natural hydraulic lime, and not a cement. The hydraulic components were also dominated by C_2S, further supporting the hydraulic lime argument. Gehlenite also forms at temperatures below 1200°C, much too low for sintering and vitrification needed for cement production. The occurrence of C_3S, which only forms at over 1250°C, is interpreted as being due to localised "hot spots" in the kiln.

Hughes and Cuthbert (2000) discuss the petrographic analysis of 12th and 13th Century mortars from the West of Scotland, drawing conclusions for the practical formulation of replacement mortars. The binder of these mortars is observed to be locally inhomogeneous, with variations in density over a small scale. Extensive porosity has developed in some parts of the mortars through the dissolution of carbonate binder material. This porosity is delineated by secondary re-crystallisation of carbonate around the pores, indicating long saturation of pores with carbonate saturated waters. These features indicate clear changes in texture of the mortars through time implying that analysis of the current composition of the mortar, especially of the binder:aggregate ratio would furnish a result different from the original composition of the mortar. This cautions against the use of simple analysis techniques that do not consider the detailed texture of the mortar.

Hughes and Cuthbert (2000) also describe the presence of fragments of what are interpreted to be unburnt pieces of limestone in the mortars they analysed, which now act as a component of the aggregate. This allows the original raw material source for the lime-binder production to be identified and analysed. Lime inclusions are also identified in these mortars, and a clear origin in burnt limestone is suggested due to the presence of relict 'psuedomorph' carbonate textures in the inclusions.

Leslie and Hughes (2001) describe the occurrence and characteristics of clinker grains in early 19th Century mortars from Charlestown, Fife. These grains have a mineralogy consistent with lime production, with concentration of silica from hydraulic lime production into these grains, but also higher kiln temperatures than would normally be expected. They also indicate that the material from Charlestown was not ground prior to use, but that the clinker may have contributed some hydraulic set to the mortars, though acting primarily as an aggregate.

The application of optical petrographical techniques are a powerful means of deriving information about the composition and history of historic mortars. Their adoption in more routine analysis is advised, but not frequently adopted. In addition the application of petrographic analysis requires specialist skills and should only be attempted by properly trained personnel, who are most often geologically educated (Middendorf *et al. in press*).

4.2.3. X-ray Diffraction (XRD)

In addition to chemical and microscopical analyses, X-ray diffraction (XRD) analysis is suitable for the identification and differentiation of binders and aggregate within a mortar, if they are crystalline. For example, the differentiation of cement and natural hydraulic lime is only possible by mineralogical analyses (Gödicke-Dettmering and Strübel, 1996; Callebaut *et al.* 2001). In X-ray diffraction a flat

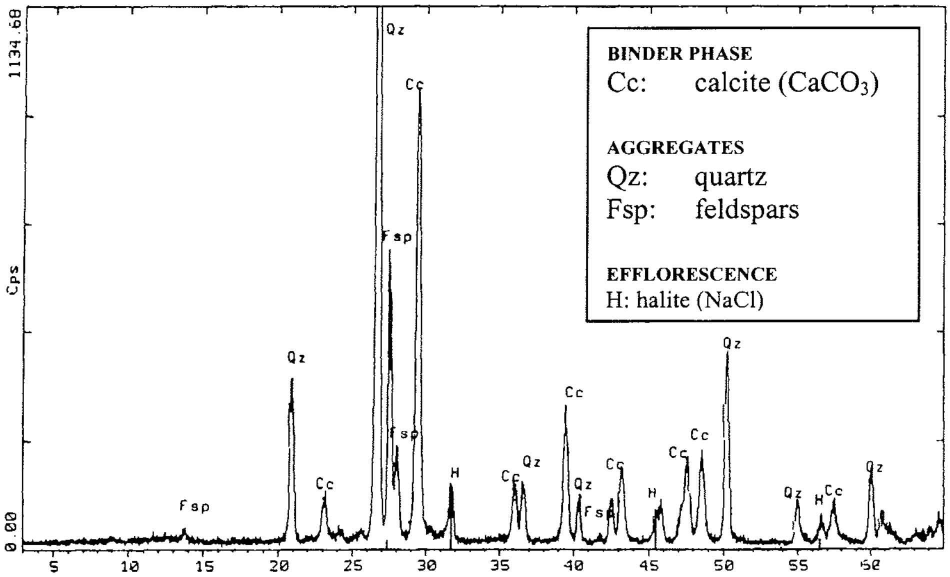


Figure 4.3 Example of an X-ray diffraction (XRD) phase diagram of a historic lime mortar (bulk sample). From Middendorf et al. (in preparation 2002).

powdered sample is exposed to a collimated X-ray beam which interacts with the sample and is diffracted back from the sample to a detector. The pattern of intensity of refraction with changing angle of incidence of the X-ray beam is characteristic of the mineralogy of the sample. In this way the composition of a mortar can be determined. However, XRD is a bulk material analysis method. It can give no information on the spatial interrelationships of mortar components, or their structure. Figure 4.3 gives an example of an X-ray diffractogram of a historic mortar.

Lewin (1981) makes use of x-ray diffraction to confirm the identification of mineral phases in specially prepared mortars in conjunction with SEM studies of microstructure and crystal shape. He states that XRD provides more useful information than that derived from chemical, elemental analysis by X-ray Fluorescence or wet chemical methods, which tend to mask the individual contributions from different minerals. However, XRD cannot cope with the identification of amorphous components, commonly C-S-H components of hydraulic mortars.

Marchese (1980) studied the composition of non-hydraulic lime mortars forming the substrate of some 12th Century Mosaics in Salerno, Italy. Thermal analysis of the materials clearly identified the presence of $Ca(OH)_2$ and $CaCO_3$. However, the XRD analysis failed to pick out $Ca(OH)_2$. This discrepancy was attributed to the presence of $Ca(OH)_2$ in an amorphous form. This emphasises the need for phases to be crystalline for XRD to identify them and the importance of not relying on one analysis technique. The application of XRD in combination with thermal analysis in this case revealed more about the nature of the constituents of the mortar than would have been apparent from using only one technique of analysis.

Rayment and Pettifer (1987) in their study of the mortars from Hadrian's Wall, used XRD to positively identify C-S-H phases and Wollastonite (CS) in the mortars. Moropoulou *et al.* (2000) used XRD to study the mineral constituents of historic mortars from Rhodes, identifying mainly the mineralogy of the aggregates (better done visually by optical microscopy), the calcite binder and some hydraulic phases in some mortars. Franzini (2000) used XRD to complement electron microprobe and XRF chemical analyses of mortars from Pisa. They noted that the binders are composed of Ca-Carbonate phases, calcite, aragonite and vaterite. More than 100 ancient mortars from Italy, Greece, Crete and Cyprus of age ranging from 1400-3000 years, were examined using XRD by Langton and Roy (1984). They identified that calcite is the predominant crystalline phase in these ancient mortars. They also detected smaller quantities of hydrogarnet and analcime in pozzolanic Roman mortars. Amorphous C-S-H phases were also detected even though the samples were up to 3000 years old.

Middendorf and Knöfel (1991) also place XRD analysis within the context of a larger scheme for the analysis of historic mortars, to complement chemical analysis. They determined the composition of the aggregate from mortars in Northern Germany and also the presence of salts. Gulec and Tulun (1997) present the results of the analysis of Roman, Byzantine and Ottoman mortars from Anatolia, using XRD, as well as optical petrography, aggregate grading and porosity measurements. The XRD analysis of the bulk mortars identified the phases present in the aggregate, the carbonate nature of the binder and some gypsum which was interpreted to be due to atmospheric pollution effects. Alvarez *et al.* (2000) perform a similar analysis on 13th Century mortars from Pamplona in Spain, identifying a lime (calcium carbonate) binder and a silica rich aggregate, and in combination with chemical analysis and thermal analysis, deriving the original mix proportions of the mortars. XRD is also applied to the detection of crystallised alteration products that can be the cause of damaging reaction in mortars or cementitious binders, for example ettringite (Martinet & Quenee 2000) or thaumasite (Collepardi 1999).

In summary XRD is a very useful technique for the determination of the crystalline, mineralogical components in a mortar. It is limited by being a bulk technique that does not reveal anything about texture or spatial distribution of the components in a mortar. However, it is quick and relatively inexpensive to use, but is best used together with other supporting chemical and textural analysis techniques.

4.2.4. Infra Red Spectrometry

This method of analysis relies on the interaction between applied infrared radiation and the molecules in compounds. Bonds between atoms have distinctive geometrys and natural states of rotation and vibration. Incident infrared radiation will excite these vibrations and rotations when a critical wavelength is reached that can impart energy to the bond. At this point the atomic bond that is being excited will absorb that wavelength of infrared. If the sample is placed between the source of infrared radiation and a detector, these times of absorption of the infrared radiation can be recorded as reduced intensity and can be related to specific types of atomic bonds characteristic of particular functional groups in compounds, for example CO_3 in carbonates. Infrared spectrometry is therefore suitable for the identification of materials and the study of chemical structure and the nature of inter-atomic bonds. For our purposes we are solely interested in the identification of mortar materials, primarily in the binder, and the possibilities for the quantification of their abundance.

Lee *et al.* (1997) demonstrate the potential for the use of infrared spectrometry in the determination of the Calcium Carbonate content of limestones. The benefits they showed are its ready calibration to other methods and the reduction in time and resources compared to conventional wet chemical methods. In another experimental study Hakanen and Koskikallio (1982) demonstrate that infrared spectrometry can be applied to the quantification of artificial mixtures of aragonite and calcite with an accuracy of 3%, and Featherstone *et al.* (1984) quantified the content of carbonate in artificial carbonated apatites (Calcium phosphate) to a high level of certainty, using the ratios of transmission, absorption and extinction of carbonate correlated against known carbonate content. Though not directly concerned with the identification or quantification of materials in historic mortars, these studies demonstrate the potential for accurate determination of material quantities of carbonates using infrared spectrometry. In addition the technique appears relatively easy to use, requires small samples and is not time consuming.

Studies of historic mortars that employ infrared spectrometry rarely use the technique in isolation. Most commonly it is used in combination with X-ray diffraction (XRD), Thermogravimetry (TG) or Differential Thermal Analysis (DTA). For example Paama *et al.* (1998) studied mortars from a 13-14th Century church in Estonia. Simultaneous TGA and the analysis of the evolved gas by Fourier Transform Infrared Spectrometry (FTIR) was used in addition to Inductively Coupled Plasma Atomic Emmission Spectrometry (ICP-AES) to characterise the materials in order to supply information for the specification of replacement restoration mortars. The combination of FTIR with TG confirms the evolution of water and CO_2 from the samples. In two papers Luxan *et al.* (1995, 1996) describe a comprehensive analysis scheme which includes infrared spectrometry. Gypsum was easily identified and confirmed by other techniques such as XRD and SEM analysis. In the later paper (1996) analysis of lime mortars from the Dominican Republic show that carbonate is easily recognised. Silicates (as SiO_4) were revealed on the spectra, and the presence of CSH fibres confirmed by SEM investigations. The potential for recognising hydraulic components therefore also exists. Furthermore, Luxan *et al.* also identify the presence of organic compounds, which the infrared method is particularly suited to identifying.

Indeed, Doménech Carbo *et al.* (1996) describe the application of FTIR to the study of the materials used in 16th-18th Century wall and canvas painting in Spain. Varnishes, pigments and other organic based substances can be identified easily. In addition the inorganic grounds of wall paintings are amenable to analysis also, with quartz calcite, gypsum and anhydrite being revealed. The FTIR technique can use very small samples of less than 0.5mg with an area of less than $0.5mm^2$, making it well suited to the study of valuable objects or indeed building fabric.

In another study, Appolonia (1995) applied infrared analysis to the study of a late plaster coating covering 11th Century frescoes in Aosta Cathedral, Northern Italy. A large scale study of the material was mounted to facilitate its effective removal. Over 100 samples were taken and analysed by microdiffraction, and FTIR with photoacoustic equipment. Both these techniques allowed the non-destructive measurement of the samples on exterior and interior surfaces. The plaster was found to be lime with gypsum that increased in quantity to the exterior surface.

Finally, Bruni *et al.* (1998) was able to distinguish between three mortar types containing calcite, magnesite and hydromagnesite using FTIR and micro-FTIR analysis as part of a scheme with thermal analysis (TG and DSC) and chemical analysis using ICP. Bruni concluded that it was possible to characterise all of these minerals successfully with FTIR, though no attempt was made at quantification.

4.2.5. Thermal Analysis (DTA and TGA, also known as TG)

Thermal analysis can be applied to mortars using three basic techniques, Thermogravimetry (TG), Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC). Each method, though having its distinct features gives approximately the same information, being based on the physical transformations that compounds experience on being heated in controlled conditions.

Thermogravimetry measures the weight loss in a sample as it is heated. Weight loss during heating can be related to specific physical decompositions in the materials that are due to the effects of increasing temperature. For example gypsum can be recognised by weight loss between 120 and 200°C as it loses water and transforms to anhydrite.

Differential Thermal Analysis (DTA) and Differential Scanning Calorimetry (DSC), are the most useful, and most used methods. In DTA a graph is continuously plotted during heating that shows the temperature difference between the sample and an inert standard of aluminium, which is heated at the same rate at the same time. Endothermic peaks are recorded when the standard continues to increase in temperature and the sample does not. At these times the sample is absorbing heat energy and using it to drive decomposition or a mineralogical transformation, usually the loss of chemically bound elements, for example water from Gypsum or carbon dioxide from

Calcite and Dolomite. The endothermic or exothermic transitions are characteristic of particular minerals, which can be identified and quantified using DTA.

Differential Scanning Calorimetry (DSC) follows the same basic principle as DTA. Whereas temperature differences are measured in DTA, during heating using DSC, energy is added to maintain the sample and the reference material at the same temperature. This energy use is recorded and used as a measure of the calorific value of the thermal transitions that the sample experiences (Willard *et al.* 1981). DTA and DSC possess another advantage over TG in the identification of minerals in mortars in that they are capable of resolving polymorphic transformations in compounds that do not involve weight loss. An example of this is given by Newton and Sharp (1987) where quartz aggregate in plasters undergoes the transition from β-quartz to α-quartz at 573°C, something they suggest could be usefully employed as an internal temperature calibration.

Adams *et al.* (1992, 1993 and 1998) use thermal analysis as the primary technique in the characterisation of historic mortars and as the basis for the study of the formulation and behaviour of replacement mortars, presumably for use in repairing the historic structures in question, namely medieval cathedrals in France and England. They are also concerned with the study of the carbonation of the historic mortars. However, the results are vague. The first paper presents the most rigorous treatment of mortar characterisation, with a straightforward identification of calcite and gypsum. However, many related issues relating to sampling and sample preparation remain unclear thus lessening the utility of the results (the fraction of the mortar that was analysed is not specified).

Ellis (2000) introduces thermal analysis clearly and progressively by presenting a range of examples of analysis of historic mortars, and classifying them according to their contents, whilst acknowledging that similarities between mortars could be misleading, and hard and fast classification is near impossible given the variety of individual materials utilised historically. A range of compositions are identified, though some ambiguity is evident in the attribution of endothermic and exothermic reactions seen in the thermographs. For example Ellis suggests that an endothermic peak between 100-130°C could be due to water loss from CSH or from clays. The presence of Calcium Silicates was suggested by correlation with chemical analysis which revealed a proportion of Silicon, though it is not made clear if this was derived from the binder or from the bulk mortar. Ellis concludes by cautioning against the use of thermal analysis in isolation and suggesting the use of other chemical and mineralogical analysis methods to confirm identifications.

Ellis (op. cit.) however, does not deal with the analysis of magnesian, or dolomitic, mortars. Bruni *et al.* (1998) studied internal and external renderings ageing from the 6th - 17th century, using TG and DSC. They were able to identify calcite ($CaCO_3$ 650°C), brucite (also called hydromagnesite $Mg(OH)_2$ 350-400°C) and magnesite ($MgCO_3$ 480-500°C).

Newton and Sharp (1987, 1987i) report in their study of renaissance plaster from the Sheffield area of England a majority of magnesian binders in their sample set. They successfully identify brucite and magnesite as well as calcite by XRD. Further study using DTA and TG confirmed this identification, but with added clarity. However, some uncertainty exists over the attribution of the endotherm often observed in the range 490-570°C, which could be due to either magnesite or portlandite ($Ca(OH)_2$- Calcium Hydroxide). Marchese (1980) studied 12th Century lime based mosaic substrates from the Duomo in Salerno in Italy, and had identified the endotherm at 510°C as being due to portlandite. However, XRD of the same samples failed to reveal any portlandite, but only magnesite. Newton and Sharp (op. cit.) contend that the 510°C endotherm is due to magnesite, and only found in their samples of dolomitic origin, quantified by atomic absorption spectrometry. On preparing experimental mixtures of pure magnesite and portlandite and testing them by DTA, it was found that the endotherms for magnesite and portlandite were missing, but ones for calcite and brucite were present. A reaction between the portlandite and magnesite was clearly occurring during heating to generate calcite and brucite. Newton and Sharp convincingly confirm the identification of the endotherm for magnesite at around 500°C. They also identified an important limitation in the use of thermal analysis to analyse magnesian binders that contain free uncarbonated portlandite. Paama *et al.* (1998) using TG-DTA provide identifications of brucite at 350-420°C, magnesite at 450-520°C, and also demonstrate how portlandite decomposes between 400-520°C allowing confusion with the identification of magnesite. They overcome this problem by performing parallel analysis using FTIR and ICP-AES elemental analysis.

The identification of hydraulic components in historic mortars using thermal analysis has not yet been convincingly demonstrated. The analysis of the hydraulic components of Portland Cement is however well understood (Taylor 1990). The main hydraulic clinker phases of Portland Cement C_3S and C_2S undergo phase transitions at a range of discrete temperatures from 500°C to 1425°C. This in principle permits the identification of unhydrated C_3S and C_2S in hydraulic mortars. However, C_2S, which is likely to be more common in natural hydraulic lime and mortars found in historic buildings due to the lower temperatures required for its formation, undergoes its phase transitions at temperatures in excess of 693°C (Taylor 1990). It is at approximately this temperature that calcite begins to disassociate. As calcite is generally a dominant phase in historic mortars it will tend to mask the identification of C_2S.

A significant body of mortar analysis work utilising thermal analysis as the main characterisation technique has been performed by a group of Greek and Italian researchers (Moropoulou *et al.* 1995a, 1995b, 1999, 2000a, 2000b, Bakolas *et al.* 1995, 1995, 1998). The mortars analysed age from Byzantine mortars from Crete and Istanbul to 18th Century mortars from Venice. The work is generally aimed at the characterisation of components and the elucidation of the historic technologies used in the production of the mortars. Thermal analysis is used as part of a wider scheme of analysis including petrography, SEM using Energy Dispersive X-ray analysis/spectroscopy (EDX or EDS), XRD and even Transmission Electron Microscopy (TEM). Moropoulou *et al.* (1995a) introduces a convenient form of classification of mortar which is used again in several papers (Moropoulou *et al.* 1995b, 1999, 2000a, 2000b Bakolas *et al.* 1995, 1995, 1998). The classification appears to successfully distinguish between mortars with differing hydraulicities (see figure 7 in Moropoulou *et al.* 1995a). To do this they use the weight loss from a sample during Thermogravimetry between 200-600°C to represent all the structurally bound water in hydraulic components. They plot this figure against the weight loss due to the decomposition of calcite, both figures transformed to percentages. They were able to define domains on the graphs for crushed brick mortars, cements and hot lime mortars though the best distinction was between these hydraulic types and the non-hydraulic lime mortars. However, these workers do not demonstrate that their apparent assumption of hydraulic water content being liberated from the mortars between 200-600°C is based on experimental evidence. Nevertheless, the fact that the ratio plots appear to distinguish the different types of mortar perhaps is a fair vindication of their approach.

4.2.6. Microstructure studies using Scanning Electron Microscopy

The Scanning Electron Microscope (SEM) revolutionised the study of materials. It enables very small structures to be imaged directly (down to 10's or 100's of nanometres. 1 nanometre = 1 x 10^{-9}m, or 1 millionth of a millimetre). Its application to mortar studies mostly involves the qualitative and quantitative characterisation of components of mortars and their textures of occurrence. It can be applied to rough fractured pieces of mortar, to look at the three-dimensional structures or on polished two dimensional surfaces of thin-sections or simple cut blocks. To reiterate, it is used for the characterisation of the morphologies and textural interrelationships of mortar components, including carbonates and hydrates (their nature, form and structure) in the binder, and the identification of alteration phases (Groot *et al.* 2000). Only very small samples are required for the three-dimensional analyses, perhaps only 1 gram, with only a matchbox sized piece of mortar required for thin-section manufacture.

Indeed, Scanning Electron Microscopy is amongst the most commonly applied techniques for the analysis of historic mortars, though its relatively high cost (£150/hour in 2002) means that its use is mostly limited to specialist research centres in universities or national building research institutes. The reason for its popularity is probably related to its ease of application, ease of sample preparation, the attractiveness of the images that can be produced (see Fig. 4.2) and the small sample size required. These last two points are perhaps the most important, and bring the method in for some criticism. Representivity is a major requirement for the interpretation of micro-structural features, though this is very difficult to achieve on such a small scale (Goins 2000) and the eye is often drawn to 'interesting' details which may not be very representative of the whole. Again, when planning mortar analysis the use of the SEM must be carefully justified and the information that it will furnish carefully determined.

The studies of Stewart *et al.* (1996) and Hughes *et al.* (1998), funded initially by Historic Scotland in 1995-96, present the first qualitative evaluation of the microstructures in historic Scottish lime mortars using the SEM. Hughes *et al.* (1998) were able to suggest a broad classification of the microstructures seen into four types, though the relationships of these to external influencing factors, such as raw materials, workmanship, interactions with other masonry materials and environment remain unclear. The mortars studied, from the east and west of Scotland ageing from the 12th to the 16th Century were all fully carbonated pure lime mortars, with no evidence of hydraulic components. They consist of a range of particles generally 5 microns (1 x 10^{-6}m) across with widely varying morphologies from blocky angular fragments to amorphous pastes.

Hughes and Cuthbert (2000) use the SEM to directly image recrystallised carbonate within a 12th Century mortar from the west of Scotland. They were able to clarify the mineralogy of the material from its habit (shape) and cleavage angles in bladed crystals growing into pore spaces.

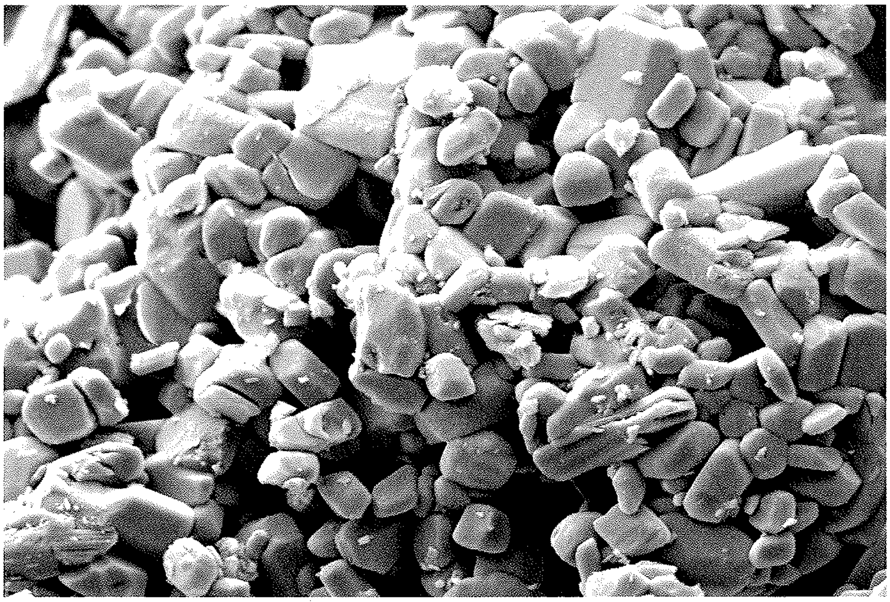


Figure 4.4: Historic Gypsum mortar. The morphology of the crystals visible aid in the identification of gypsum. Field of view 145 microns (0.145mm). Picture B. Middendorf, GH Kassel, Germany.

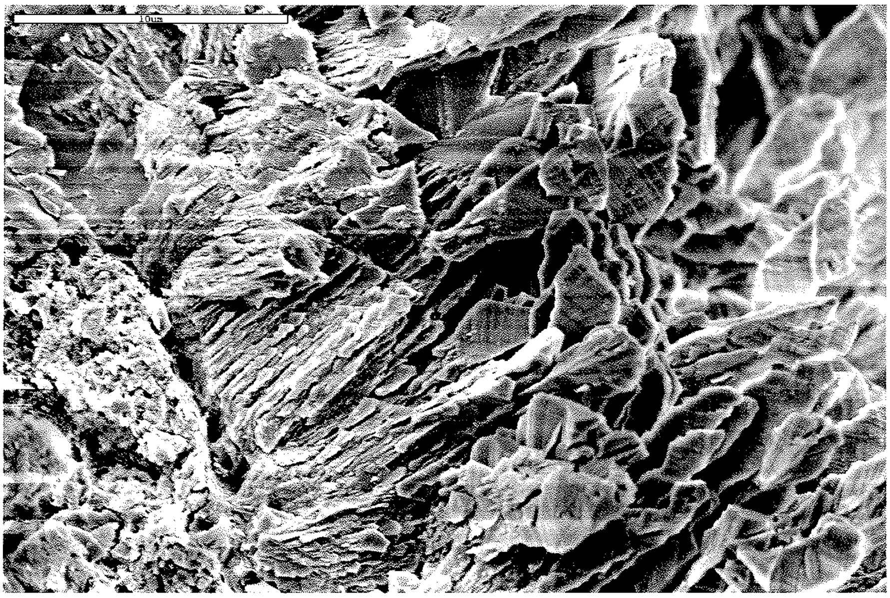


Figure 4.5: A three-dimensional view of an isopachus calcite pore lining in a 12th Century mortar from Inverlochy Castle. Binder substrate is to the left, followed by a coalesced layer of perpendicular crystal growth, topped by open bladed crystal growth to the right. This is a detailed view of calcite crystals that have grown within pore spaces within an historic mortar, the cause of what is commonly referred to as "autogeneous healing" in old lime mortars. Field of view about 30 microns (0.03 mm).

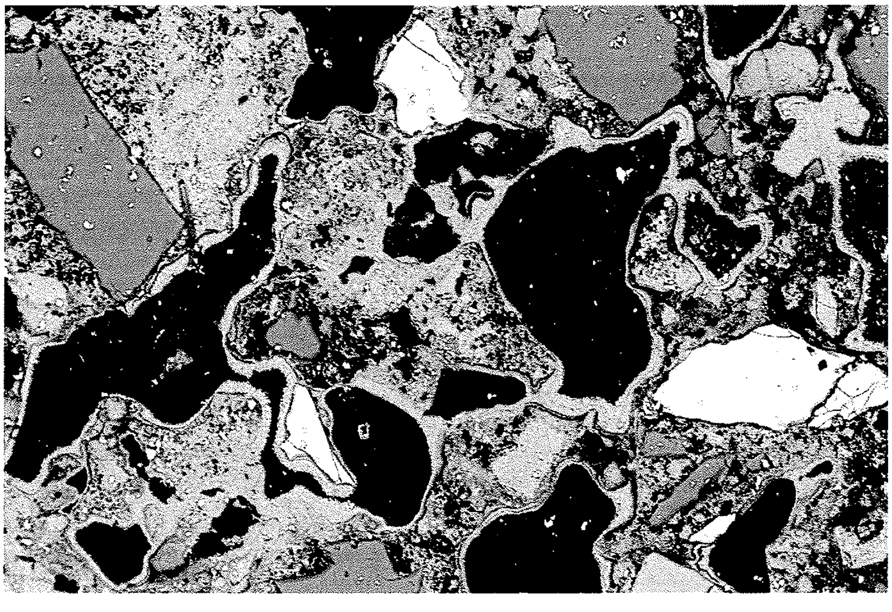


Figure 4.6: Scanning electron microscope (SEM), Back-Scattered Electron (BSE) image showing enhanced porosity (Black) in Inverlochy Castle mortar. Pores lined by recrystallised calcite fringe, seen here in light grey. Field of view 800 microns (0.8 mm). Such images depend on the contrast in atomic number and density of the materials in the sample. Investigation of materials using BSE imaging requires a flat polished surface to be prepared on a cross-section cut through the sample.

Hughes *et al.* (1998) and Hughes and Cuthbert (2000) also make use of Back-Scattered Electron (BSE) imaging in the SEM on flat polished thin-sections. This technique is commonly used to study the microstructure of cementitious binders (Scrivener and Pratt 1984, St. John *et al.* 1998, Diamond 1999, Hooton and Brown 2001, Famy *et al.* 2001). The advantage of BSE imaging is that the grey level contrast in the images is based on the composition of the phases present within the material. This technique can therefore pick out compositional variations and also be combined with chemical element analysis, that can be mapped across the sample, over a large area or in a single spot. The combination of these two is very powerful and can aid in the identification of hydraulic components (e.g. Callebaut *et al.* 2001) and hydration products in binders. Rayment and Pettifer (1987) apply BSE imaging to the characterisation of the hydraulic components of mortars from Hadrian's Wall and the reaction rims around chert grains in the aggregate.

Lewin (1981), used the SEM to study the nature of a range of specially prepared mortars containing lime, hydraulic lime, pozzolana, cement and normal aggregate. Several distinctive features of these could be identified by SEM, and the presence of hydraulic components in particular were identified. However, Lewin also used XRD to study the mortars and concluded that neither technique alone could provide an adequate characterisation of the mortars. He states that SEM reveals the "sizes, shapes, and textures of the internal structures in the mortar..., but morphology is not an unambiguous key to composition". He recommended an integrated analysis of which SEM plays a key role.

A new development in SEM technology, the Environmental Scanning Electron Microscope has recently become available to researchers. This version of the SEM allow samples to be viewed under wet conditions, controlled gas atmospheres and also during heat to up to 1000°C. This therefore permits the study, in real time of dynamic processes in materials, for example the hydration of quicklime, the calcination of stone or the carbonation of lime mortars (Allen *et al.* 2000, Radonjic *et al.* 2001).

4.2.7. Electron Microbeam analysis

Another method of analysing the chemical composition of a material is to use electron beam methods. In these a beam of electrons is directed through a vacuum onto the sample to be analysed. Once hit by the electrons the material will emit x-ray radiation of varying wavelengths dependant on the elements present. The intensity of this radiation can be detected and its intensity related to the proportions of elements within the sample at the point the electron beam hits. Specialist analytical equipment exists called the Electron Microprobe, but the measurement technique is now available on all Scanning Electron Microscopes, commonly referred to as EDS, or EDX - Energy Dispersive X-ray analysis. The method is routinely used to confirm the identification of mineral species in historic mortar studies, especially when imaging of microstructures is taking place (e.g. Callebaut *et al.* 2001).

However, quantitative analysis can also be applied to very accurately measure the chemical composition of mortar constituents, if a flat polished surface is prepared, in a polished thin-section or polished block. For example, Rayment and Pettifer (1987) applied the method to the characterisation of hydraulic phases (C-S-H) and found that the ratios of CaO: SiO_2 in these phases was similar to those found in modern C-S-H phases, consistent with formation from β-C_2S.

Franzini *et al.* (1999) studied the composition of 90 samples from Pisa to compare them with ancient pozzolanic mortars. They used EDX analysis to characterise the composition of the binder. They discovered pure non-hydraulic lime and hydraulic binders. They analysed the composition of the lime inclusions in both and found that the composition was approximately the same. Interpreting this as the original composition of the binder, being unmixed 'clots' in the mortar, it is suggested that the formation of the apparently hydraulic binder is due to the addition of a reactive siliceous aggregate, probably a diatomaceous earth. This will have acted as a pozzolan, even though no obvious pozzolan is identified in the mortar.

In a later study Franzini *et al.* (2000) continue their investigations of mortar from Pisa. Normative calculations of the chemical composition of the binders are performed combining data from bulk analysis by X-ray Flourescence analysis, and the weight fractions of binder and aggregate, to derive volatile CO2 and H_2O contents of the mortars. The mortars consist of crystalline calcium carbonate mixed with an amorphous calcium silicate hydrate phase.

Electron Microbeam techniques promise detailed and precise chemical analysis of mortars, that can be related to textures and component assemblages and reactions between them. Combined with SEM studies and BSE imaging it is another powerful method that has not yet seen much use in historic mortar studies.

4.2.8. Physical and Mechanical testing

Physical testing of mortars predominantly encompasses the determination of properties such as the pore structure characteristics (capillarity, density, shrinkage, porosity, permeability, water absorption etc.) and also thermal expansion characteristics of mortar. Mechanical testing includes measuring the strength characteristics of the mortar, the modulus of elasticity, adhesion bond and surface hardness, for example. Charola & Henriques (2000) considered that the characterisation of the physical and mechanical properties may be sufficient for the compatible matching of mortars, and that the characterisation of composition is not necessary (see below section 4.4). Physical properties, especially porosity are frequently measured, but mechanical properties are not, due to difficulties over obtaining sufficient sample (Groot *et al.* 2000).

Schouenborg *et al.*.(1993) analysed the mechanical and physical properties of mortars from three medieval churches in Sweden. In the field an adhesion test was performed by drilling a core and then recording the force required to pull the sample out. Compressive strength was measured in the laboratory, by halving the rather small samples and reassembling them with a thin layer of mortar in order to achieve the required thickness for the test. Most mortars achieved a strength of >4.5MPa, enough to satisfy Swedish Standards. Porosity was also measured by determining the weight, density and the bulk volume of the samples. The apparent density was measured by using a helium pycnometer. Frost resistance was measured along with capillary suction and drying. These tests were part of a comprehensive analysis scheme incorporating chemical and petrographic analysis.

Binda and Baronio (1988) and Baronio and Binda (1988) characterise the densities of historic mortars in Italy, and also the Initial rate of absorption in a consideration of the nature of the brick-mortar bond. Moropoulou *et al.* (2000) tested the tensile strength of historic mortars using the method of Katsaragis (1987) and Tassios *et al.* (1989). They found an inverse relationship between this property and the ratio of CO_2:structurally bound water. They found that as hydraulicity increases (with decreasing CO_2 levels) the tensile strength increases.

Porosity can be measured in mortars using a variety of different methods ranging from the relatively simple to the complex and instrumental (Thomson *et al.* 2002, *in preparation*). These can include indirect methods such as simple saturation density methods, mercury intrusion porosimetry, and indirect petrographic methods using automated image analysis. Some indirect methods such as mercury intrusion porosimetry are considered by some to be inappropriate for use on lime-based mortars as they damage the texture of the mortar and lead to incorrect estimations of porosity. See also section 5.3.1. for more information about mortar porosity.

Válek *et al.*, (2000), experimented with the application of in-situ gas permeability measurements on mortar and sandstone. This is a non-destructive test, where a probe is sealed by pressure against the surface of interest and nitrogen gas pumped under pressure into the material under test. The gas flow rate into the material and pressure are recorded once a steady state is reached and can be used to calculate the permeability. They were able to measure the permeability of sandstone with reasonable repeatability, but the variability of the measurements was high due to variability in the cross-bedded sandstone that was tested. This was attributed to grain size variations in the sandstone and the moisture content of the stone. Testing of laboratory prepared lime mortars revealed a major control of results was due to surface finish, more than the effects of curing conditions. Válek *et al.* (2000) conclude that the method was considered to have some use in the determination of the compatibility of original and new materials.

4.3. General Analysis Schemes

Many attempts have been made to systemise the analysis of mortars from historic buildings and to establish protocols for comparison of results derived from different analyses. (For example Charola *et al.* 1986, Middendorf and Knöfel 1991, 1998, Dupas and Charola 1986, Goins 2000, van Balen *et al.* 2000, Martinet and Quenee 2000, Callebaut *et al.* 1999, 2000).

Middendorf and Knöfel (1991 and 1998) worked on the formulation of a number of flow charts to assist in mineralogical, chemical, and physical characterisation. These, as well as other schemes, can be applied as an aid to analysis - not as proscriptive schemes. These charts give a comprehensive idea of the possibilities for characterisation in the different fields as described above, and also put useful constraints upon the sample requirements and sample preparation needed. They clarify the potential pathways for analysis, allowing analysts to chose that appropriate for their purposes, without precluding the later use of other methods. The conservative use of sample should be encouraged, if the range of potential analysis is understood, reducing the need for later re-sampling on-site (Van Hees 2000).

Callebaut (2000) also presents a similar detailed procedure combining a full range of mineralogical and chemical techniques. He emphasises how early analysis schemes concentrated mostly on wet chemical methods (Jedrzejewska 1960, Cliver 1974, Dupas 1981), whereas more modern approaches incorporate more mineralogical and petrographic methods. They are even beginning to incorporate more physical characterisation as well as compositional studies, as the properties needed for the specification of new or replacement mortars develops its concept of compatibility. Physical characterisation for strength and porosity for example are becoming more common (Baronio and Binda 1991, Knöfel and Schubert 1999, Van Balen 1999). No standard combination of methods, and as we have seen, no standardisation amongst methods exists at present. This presents aproblem of comparability between results and between laboratories, also leading to some confusion about the important parameters that should be measured.

4.4. Characterisation with a view to repair

Groot *et al.* (2000) discuss the need for the specification of parameters that should be characterised in order to better formulate a repair mortar that will be compatible with existing building fabric. The activities of the RILEM TC-167COM "Characterisation of Old Mortars with Respect to their Repair" have been aimed at clarifying this, and the methods needed for characterisation. The publication of the workshop proceedings from the committee in 2000 (Bartos *et al.*, 2000), contributed significantly to the debate.

The relevance of detailed knowledge regarding the characterisation of old mortars is considered by Leslie and Gibbons (2000). One of the most important factors in the analysis of old mortars is an understanding of the surrounding building structure and conditions. The same applies to the existing function of the mortar. An interesting example in this respect is the differences in function of 'sealing' mortars in dry stone build with no mechanical function (Maxwell 2000) and thick joint masonry (Byzantine, Baronio and Binda, 2000) that have a significant mechanical function.

Leslie and Gibbons also observed that the data that are relevant to the requirements of building conservation are in general: the hydraulicity of the binder, the relative weights of binder and aggregate and the aggregate grading in order to identify the necessary components to produce a compatible mortar. This information can be obtained through simple examination by eye and binocular microscope coupled with acid dissolution and aggregate separation, and does not necessarily require expensive analyses.

More emphasis on porosity and strength characterisation was advocated by Charola & Henriques (2000). It would appear that the identification of actual hydraulic components may not be necessary for either the characterisation of the mortar or the development of a successful formulation for its replacement. Determination of porosity characteristics related to strength may serve as a more important guideline for matching repair mortars to existing ones in historic structures, than detailed knowledge of hydraulic components. The work of Válek *et al.* (2000) on the in-situ gas permeability of masonry may relate a porosity-related property indirectly to strength and hydraulic properties. This approach underlines the importance of the characterisation of moisture condition and behaviour within masonry. However, again it appears that a combination of methods is perhaps more appropriate for characterisation.

The notion of "external requirements" having an influence on both the analysis of historic mortars and the formulation of their replacements is now being discussed, though has not reached publication at the time of writing (early 2002). What is meant is the influence of issues such as conservation philosophy, authenticity (as defined by the Nara Document, 1995) cost etc. These are issues "external" to the technical analysis and characterisation of the mortars that will affect how they are sampled, analysed and reformulated (Van Balen *et al.* 2002, in preparation). However, this is contentious and some believe best left to architects or building conservators, not scientists.

5 DESIGN AND DEVELOPMENT OF NEW MORTARS

This section reviews current efforts in the development of mortars intended for the conservation of historic buildings. It concerns the design and testing of new mortars, and the final steps of the whole procedure before the ultimate application of conservation treatment. The previous chapter detailed evaluation and analysis of historic mortars and masonry where all the information was collected with an apparent aim to specify replacements. In this chapter the extent to which this relationship affects the development of new mortars is explored further. The latest results of practical, research, theoretical and experimental work are presented and assessed, together with a review of novel means of testing. Much of the following text also relates closely to the previous discussion on compatibility of materials in historic buildings.

5.1. Specification and performance of new mortars

Whenever a new repair mortar is to be designed, the 'compatibility testing approach' should be used. It ensures that the new mortar is compatible with the original one. Gonçalves (1998) described a methodology used for the design of a new compatible render, which typifies the latest ideas behind the compatible design of new mortars. It stemmed from the definition of compatibility which stated that a new material should fulfil all the functions required from it and should not introduce any new damaging actions. This definition of compatibility describes general requirements which have to be interpreted for its practical application. It has to be expressed with regard to the particular conservation treatment, materials and conditions. In general, it is known as specifications for new repair mortar. For example, one of the compatibility specifications used by Gonçalves (1998) was that the new render should 'not block the passage of the water vapour that circulates due to the gradient of water vapour pressure between the interior and the exterior of the building, by retaining it inside the wall'.

The specifications for new mortars should reflect their nature. They should be based on scientific results from research into material properties (Von Konow 1998). A number of specification criteria are usually defined. In order to find the right material which complies with these particular specifications, appropriate testing methods have to be determined. Standardised and approved testing methods are usually utilised, but new tests should be apparently employed as well, since many standard tests are not suitable for testing traditional materials (Charola and Henriques 1999).

The method of testing, whether based on composition or performance, should be clearly identified. Confusion between these two may later lead to problems (Henriques and Charola 1996). Both methods are based on a similar procedure which extends from examination of historic materials to testing and selection of the adequate mortar. The result, the new repair mortar described by its performance characteristics, should in both cases be compatible with original materials. On the other hand, the difference between these two approaches is the range of criteria on which the materials are compared.

Water vapour permeability
Capillarity
Capability for impermeabilisation
PH and soluble salts content (chlorides, sulphates and alkalis)
Adherence to support
Resistance to cracking
Resistance to impact of a round body
Resistance of renders to salt crystallisation
Artificial ageing tests – heat/rain, hot/cold, freeze/thaw

Table 5.1: Example of some performance characteristics to be measured for new renders according to Gonçalves (1998).

Criteria and tests for selection of new compatible mortar have been the subject of research for some time now (Peroni *et al.* 1981, Rossi-Doria 1986). Peroni *et al.* (1981) recognised the importance of defining technical criteria in order to ensure a more appropriate selection of mortars for conservation purposes. The authors suggested a number of tests which should be accomplished to describe a performance of mortars. They also imposed certain limits which should ensure compatibility of mortars. These limits were designed to inhibit the known cases of masonry decay, i.e. high soluble salt content, or too high compressive strength. To summarise the procedure from the literature above, the best way to design a new compatible mortar seems to be to determine the properties relevant to potential damage of the host masonry and consequently ensure that the new mortar fits within the range of these imposed limits.

Workability	Mortars should have optimum workability
Setting time	Three days maximum, although for some applications ten days may be tolerated
Compressive strength	New mortars should not be much stronger than the ones used in the old masonry (0.5 – 3.0 MPa is advisable).
Flexural strength	It is desirable for this to be reasonably large but not exceedingly so (0.4 – 2.5 MPa)
Modulus	There is no exact range recommended
Porosity	Minimum 20% with at least 65% above 0.1μ
Water absorption	Important factor but no range was suggested
Water vapour permeability	Minimum value may be desirable but no exact range was suggested
Alkaline elements	As low as possible. 8mg/kg might be reasonable.

Table 5.2: General criteria for selection of repair mortars according to Peroni et al. (1981)

The successful design of a compatible mortar lies in the ability to characterise properties of mortars and a selection of appropriate tests. Standard tests are often designed for testing modern materials only and therefore they are not relevant to traditional materials. There is a need to study and modify these tests, and it has been described during a conference 'The Use of and Need of Preservation Standards in Architectural Conservation' (Sickels-Taves (ed.) 1999). On the other hand, the selection of appropriate tests is more difficult as it is not certain what is a measure of quality of mortar in general. There is not a single characteristic that would be able to describe compatibility and a number of tests have to be used instead. However, how many tests are needed and their hierarchy have not yet been satisfactorily defined. Therefore, the general list of properties to be tested can extend to a large number of tests in order to cover all possible sources of damage. Usually, compressive strength, porosity, permeability, composition, thermal expansion etc. are more or less relevant depending on the particular case. However, it should be understood that every conservation project has slightly different requirements and therefore different criteria for selection of a new mortar.

Carefully defined criteria for the selection of a new mortar, based on the understanding of original material and required remedial actions, should lead to the selection of an appropriate material. However, there is a need for more relevant tests which would deal directly with the problems related to the applications of new mortars. An example of the development of more appropriate tests can be studied in a work of Veiga and Carvalho (1998) who compared performance of lime, cement and lime gauged with cement mortars used for rendering. The mortars were compared on a basis related to their ability not to contribute to any further damage. The requirements from the new mortar were as follows:

- not to transmit any high stress to the substrate
- not to retain water within the construction
- not to have a high salt content.

Judged by these criteria, the lime-based mortar was considered to have the most suitable behaviour. On the other hand, the lime mortar failed the freeze/thaw testing. If the mortar had been evaluated according to durability represented by the freeze/thaw testing, then it would not have been the most suitable. This fact illustrates the importance of selection criteria.

5.1.1. Examples of design of new mortars

The following are three examples of how compatibility and design of new repair mortars have been approached.

(i) Papayianni *et al.* (2000) suggested designing a new mortar according to its 'functional behaviour'. This means that compatibility should be measured by properties characterising the functions of mortar in the structure. According to the authors (op. cit.) the characteristics were as follows:

- colour and surface structure
- strength, elasticity and deformability
- porosity and porosity properties
- coefficient of thermal dilation

First of all, historic mortars were analysed (microscopic examination, aggregate grading, porosity, compressive strength, chemical composition and soluble salts). All the historic mortars except one appeared to be lime-based.

Different mortar mixes were designed correspondingly to cover the variety of mortars present in the different parts of the structure. The design of the new mortar was controlled mainly by composition (binder and filler proportions) combined with porosity and strength. Colour of the mix was adjusted by the colour of aggregate. Porosity and compressive strength were tested to confirm the compatibility of mortars. Although the strength of the new mortars was slightly higher than that of the historic mortars the authors concluded that the mortars are compatible. No further evaluation of the performance was carried out after the application.

It should be noted that the materials used in the composition of new mortars were not of the same provenance as that contained by the original mortar and a white cement was even added into some mixes. Although the design seemed to be based predominantly on composition, it did not try to copy the composition of the original mortar. Rather, it considered the general properties which these original mortars possessed. On the other hand, the paper did not explain why and how relevant the tested properties (such as the compressive strength and porosity) were to compatibility. No exact specifications or criteria that would relate to the historic masonry, other than the general ones mentioned above, were specified. Although the design was not based purely on composition or performance parameters, it represented the common approach towards it. The validity of the method and the compatibility of the mortars are yet to be confirmed.

(ii) Veiga and Carvalho (1998) studied the appropriateness of using lime-based mortars for renders. The aim was to compare lime, lime-cement and cement mortars in relation to their application for conservation projects. Testing procedures were developed to describe the most crucial characteristics relevant to the compatibility specification of renders (Veiga and Carvalho 1998). The testing was divided into the following three main sections.

- *Evaluation of stress within the mortar and evaluation of tensile resistance.* Transmission of a stress from mortars (renders) to the substrate masonry could cause damage to the substrate. Therefore such transmission should be limited. The authors focused on stresses caused by restrained shrinkage measured by a methodology (evaluation of cracking susceptibility of renders) described by Veiga (1998). The method operates with two criteria for description of cracking susceptibility. The first criterion is a coefficient of opening of the first crack (maximum load force divided by tensile strength). The second criterion is a coefficient of resistance to cracking evolution (tensile rupture energy divided by tensile strength).
- *Evaluation of the ability to protect the wall against ingress of water.* An electric resistance of mortars was measured to determine the time taken for water to reach the substrate and consequently to dry out.
- *Evaluation of the durability concerning climatic actions.* Specimens of different renders were exposed to heat/cold, heat/rain and freeze/thaw cycles.

From the results obtained, the authors (Veiga and Carvalho 1998) concluded that forces developed due to restrained shrinkage in lime mortars are much smaller than those developed in cement mortars. Moreover, mortars based on lime were less susceptible to cracking than mortars with cement or pure cement mortars (Veiga 1998). Lime mortars were the most water permeable. The drying phase especially was much faster for lime mortars in comparison to mortars with cement. No significant degradation was observed for heat/cold and heat/rain cycles for all mortar specimens, however the freeze/thaw caused degradation of the lime mortars in a few cycles. The overall conclusion was that lime-based renders have the most suitable performance characteristics compared with mortars gauged with cement or pure cement mortars.

The comparison and subsequent selection was based on the requirement that new mortar should not cause any further damage to the original masonry. This was a simplified definition of compatibility and the mortars were studied strictly from this point. However, the paper did not have the scope to describe and verify the results on any real application, apart from the general experience of using lime-based renders. The research should be backed up with some practical results.

(iii) Van Balen *et al.* (1999) described a case study of the use of epoxy resin and fibreglass rods as binding materials for anastylosis of the late Hellenistic Nymphaeum in Turkey. The paper explained not only compatibility, but also the authenticity, retreatability and reversibility requirements on the design of joints between individual stones. The design also reflected the conditions in which the Nymphaeum is situated. It lies in a seismic zone where earthquake is one of the major threats to the historic buildings.

The use of modern material with a high mechanical strength ensured that intervention was kept to a minimum. The connection between individual building blocks was based on the installation of dowels which should resist the stresses between stones. On the other hand, the design of the dowels incorporated a limitation that the new connection between the stones should break first, before any damage is caused to the original masonry. Retreatability was considered in the design as a case of saving the stones undamaged, should an earthquake strike the building, to allow the reconstruction to be repeated.

The technical part of the project dealt mainly with a test of the epoxy joints to design a proper balance between the strength of the stone and epoxy resin. Compatibility was considered on the basis that the stone should not be damaged by mechanical stress. The epoxy resin in the joint should fail before any damage to the stone occurs. The adhesion of the epoxy resin was reduced by the addition of a filler which contained powdered limestone. Construction details of vertical and horizontal joints bridged by the fibreglass dowels were another part of the design which had an effect on the behaviour of the structure and therefore on compatibility and retreatability. The fibreglass dowels were designed to break instead of being pulled out when under an excessive load and therefore to give a limited ductility to the joints (Van Balen *et al.* 1999).

This last example illustrates that under certain circumstances a modern material (non-original) can be used in conservation. In such a case its use has to be fully compatible with the historic material, and other conservation requirements such as reversibility and/or retreatability have to be fulfilled. In this design, the compatibility of the mortar and the binding elements was considered mainly from a structural point of view. On the other hand, any potential weathering problems have not been taken into account. The paper demonstrates that the criteria for a compatible repair mortar should correspond to the conditions in which the structure is situated and which are relevant to potential damaging processes.

5.1.2. Design based on composition and/or properties

Groot *et al.* (2000) and Gonçalves (1998) described the design of new mortar as a *traditional* one, based on composition, and a *modern* one, based on properties. Groot *et al.* (2000) pointed out that there should be a mutual understanding of these two ways of design. Understanding what are the distinctions and common points of these two approaches helps to define the most important parameters when designing a new compatible mortar.

Factors Affecting the Design of New Mortar Based on Composition

The analysis of historic mortars allows a copy of original mortar to be designed and such an approach is now common in the conservation of historic masonry (e.g. Florez de la Colina 2000, 'Analysis of old mortars from ancient *agora* of Thessaloniky, Greece').

However, properties of mortars based on composition similar to the original ones are not compatible a *priori* and should be tested. Válek & Bartos (2001) pointed out that to copy properties of historic lime-based mortars, such as strength and porosity, merely by composition was very unpredictable, as there were other influences that affected the properties. The performance of lime mortars is influenced by many factors. Even the composition itself can be highly variable depending on the original limestone, burning and slaking conditions and type of aggregate (Jedrzejewska 1981). Some material researchers even suggest that there are too many influencing factors on old mortars that they are impossible to copy (Von Konow 1993). A similar conclusion was reached by Hughes *et al.* (1999) when the authors pointed out that physical changes of lime mortars caused by ageing may inhibit the determination of the original mortar composition.

When a new mortar is based on a compositional copy of the original mortar, a question of the accuracy of such a copy should be considered. In some cases the provenance of the raw mortar materials (limestone, sand, pozzolana, etc.) or exact production techniques can be very significant. From a conservation point of view it may be argued that when mortar requires a periodic maintenance and repair the traditional techniques should be employed (see Declaration of San Antonio 1996). Hughes *et al.* (2000) initiated a new research project into traditional lime burning. A newly built replica of a traditional lime kiln equipped with a monitoring system aims to research traditionally burned limestone in Scotland.

Durability and performance of non-hydraulic lime mortars are limited not only by the material itself but also by workmanship, ageing and curing conditions and stone and mortar interaction (Bartos and Lawson 1996). Because the performance of lime mortars is influenced by so many factors, the specification of repair mortar based on the composition of the historic mortar should consider information about the repair work to be carried out, the type of masonry and climate (Leslie & Gibbons 2000).

One of the most important influential factors on non-

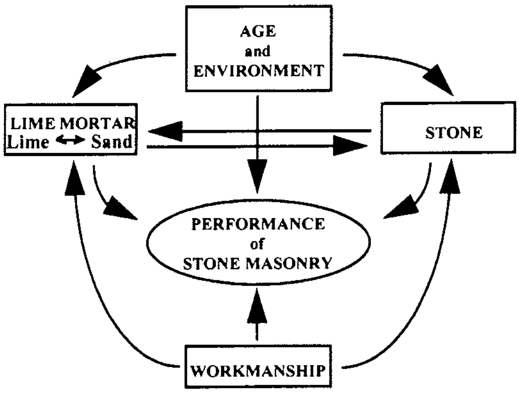


Figure 5.1 Stone and lime mortar interaction and influencing factors after Bartos and Lawson (1996).

hydraulic lime mortars is the workmanship (Gibbons 1995 TAN 1). Workmanship comprises the application method, particular constructional detailing, adequate workability, final surface finish, in-situ protection and curing. It is believed that a skilled craftsman can significantly improve the quality of repair work - 'Lime mortar consists of 99% good knowledge and 1% quality lime sand' (Holmström 1998). In the past, craftsmanship was often connected with a special formula for good quality mortar, which was in general a lime/sand mix with some organic or inorganic additives (Sickles 1987). Fisher (2000) presented his practical experience with a modern mortar, which was successfully used by a German craftsman, who utilised a number of additives to improve the quality of the mortar without studying its chemical and physical properties. The use of lime was widespread across the world and a variety of methods of mortar preparation can be compared. For example, Liu (2000) described traditional methods of lime slaking and lime mortar preparation based on oral description in China.

Factors Affecting the Design of New Mortar Based on Performance Characteristics

Design based on properties allows the use of new, non-original and non-traditional materials by guaranteeing their compatibility with the historic fabric. However, the scheme itself does not guarantee a successful compatible design. The design procedure has to be very advanced in the understanding of historic and replacement materials as well as having accurate and appropriate test methods (Gonçalves 1998). It should be remembered that since it is a relatively new subject there is a lack of experience and publications available and more of both are urgently needed to establish its correct application.

The main potential pitfalls of this design can be summarised as follows:

- The formulation of the specification criteria is the most crucial and the criteria should be relevant to each particular case.
- The selected tests should be relevant to those particular specification criteria. The tests should be reliable to describe the 'real' material characteristics under realistic conditions. Henriques & Charola (1996) compared results from testing lime mortars and pozzolanic lime mortars prepared and cured under different standard tests. The results pointed out inconsistency between various national standard tests. Some results cannot be directly compared with each other as each testing method produced different results.
- The limits or range within which the material should be compatible can sometimes be identified. Sass and Snethlage (1997) suggested a general range of criteria.
- The material selected by a comparative method from a number of different mixes is merely the best out of the available specimens. If the selection is not competitive in quality or relevant to compatibility then the selected material may not be appropriate.

5.2. Lime-based mortars

While discussing mortars and conservation of historic buildings, lime-based mortar should be mentioned as a special case on its own. This is because lime was used from ancient times up to the invention of cement as a main binder for stone and brick masonry construction throughout Europe (Malinowski 1981). Mortars used in medieval historic structures are therefore often assumed to be lime-based (Furlan 1991). Although such simplification is not valid, it demonstrates the importance of the historic and present use of lime.

Lime mortars have been used for many centuries. Their use was mastered by the day to day practice of craftsmen in prehistoric and medieval times, more or less following the rules set by Vitruvius in his classic work *Ten books on Architecture*. Their use was complemented much later on by experimental research results from the end of the 18th century by Smeaton (1793) or Vicat (1997, first published in 1837). Historic Scotland (Gibbons 1995) published a Technical Advice Note which revived the subject of the *Use of Lime Mortars*. It especially stressed the use of non-hydraulic lime mortars made of lime putty. The publication became an authoritative source of information for preparation of lime mortars used in conservation. However, the variety of composition, techniques of production and application of mortars should not be underestimated. Lynch (1998) pointed out that historic mortars were not only mortars made of lime putty or 1:3 mix. The source of limestone and the local source of sand had an influence on its composition as well as the differences in mixing and preparation. There is now increasing evidence that hot mixing and/or dry mixing (Leslie and Gibbons 2000, Callebaut *et al.* 2000, Hughes *et al.* 2001) techniques were also applied to prepare many historic mortars. Hydraulic lime mortars were also used, especially in towns and cities. In areas devoid of natural hydraulic limes, 'artificial' hydraulic limes were utilised, being non-hydraulic lime mixed with a pozzolana (Lynch 1998).

Furlan (1991) pointed out that the research into lime-based mortars has multiplied during the last two decades. Many publications suggest that new mortars for any remedial work on historic masonry should be based on the approach that pure lime mortars prepared in a traditional way show the closest performance properties to the historic masonry (e.g. Torraca, 1988). Conservation literature in many cases (e.g. Gibbons 1995) simplifies the problem of compatibility as the decision to use lime-based mortar instead of cement mortar. This results from bad experiences of using cement mortars in the past. When assessing past conservation attempts the two following points often appear:

- Firstly, the lime technology and skills were partially forgotten (Gibbons op. cit.).
- Secondly, Portland cement superseded lime because of its superiority in strength (Gibbons op. cit.) but other qualities of lime mortar were not properly considered.

These two points can be seen from a compatibility perspective. Correct application of lime mortar requires special training and care to be successful (Maxwell 1998, Gibbons 1995). Although the use of cement-based mortars was widespread, their compatibility was not considered and they were often found later to be incompatible with the original masonry material. There is a general consensus between scientific and conservation literature regarding these two points. However, the preceding review of the latest trends in conservation science indicates that a detailed acquaintance with many material properties is vital in designing a compatible material. It can be said that the better a particular material is described in terms of its properties, the better result is obtained in terms of material compatibility. A serious study of the apparent causal relationship between the use of cement-based mortars and stone and brick decay is still lacking, so statements regarding the incompatibility of cement and its rejection by the conservation practitioners are not backed by detailed quantitative evidence.

Research into mortar and concrete based on Portland Cement appears much more advanced and profound than that on lime (e.g. Hewlett 1998). The research reflects the use of cement and concrete in the modern building industry. This point illustrates the fact that there are no international lime oriented research journals (some national journals exist, e.g. The Journal of the Building Limes Forum) and research articles about lime and lime mortars are often found in journals (oriented mainly towards cement and concrete) such as *Cement and Concrete Research, Concrete International, Materials and Structures, Magazine of Concrete Research, Construction and Building Research, Brick News, Magazine of Masonry Constructions, Thermochimica Acta, Masonry International, Journal of American Ceramic Society* etc. In future it may appear that a lack of exact scientific knowledge of lime and lime mortars will be the greatest drawback in its proper use as a compatible material. A more detailed understanding of fundamental properties of new lime mortars is therefore required to back up the demands from conservation practice to use original materials (lime mortar) for conservation works (Furlan 1991, Válek 2000).

Figure 5.2: Is this mortar compatible with the masonry? Red sandstone ashlar masonry in Paisley. Cement based pointing mortar together with moisture and salt transport is often thought to cause such typical decay round the joints. The adjoining white sandstone is not suffering such extensive damage, suggesting the mortar is more compatible with that stone regardless of its composition.

Figure 5.3: Is this mortar compatible with masonry? Red sandstone masonry has weathered due to wind exposure in combination with moisture evaporation. However, the hard pointing mortar has determined the weathering pattern and helped the weathering rather than protecting the masonry. It could also be said that the combination on a masonry surface of such materials with contrasting durability (related to the bond between components and overall hardness) has resulted over time in this aesthetically problematical weathering pattern, as the sandstone weathers away more readily than the cement-based mortar.

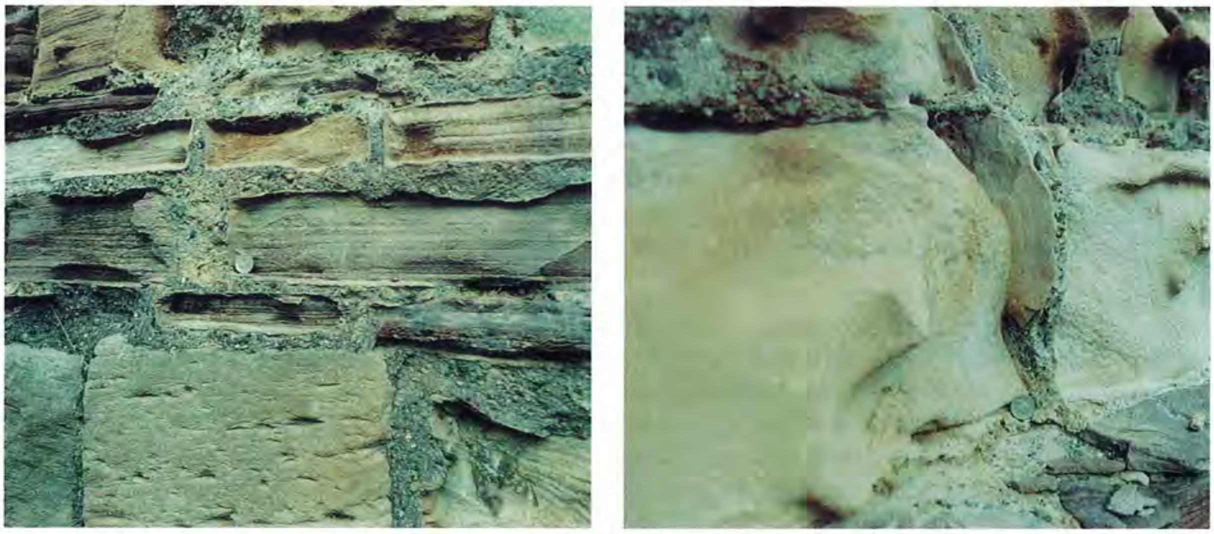


Figure 5.4: Is this mortar compatible with masonry? Cement-based mortar is a good binder and can locally support the adjacent stone to mortar joint. However, the masonry still weathers and its appearance is changing. The mortar in joints is protruding and visually expanding but stones are diminishing.

5.2.1. General composition and strength

Suter and Song (1995), in their review of historic stone masonry properties, suggested that the compressive strength of the original mortars can be expected within the range of 0.1 and 3.5 MPa. The binder to aggregate ratio was found in the range of 1:0.4 and 1:5 with the majority being 1:3 (Suter and Song 1995). Durability of lime based mortars can vary depending on their ageing conditions but examples of historic mortars surviving well over 600 years old are available worldwide. Longevity of the surviving masonry of historic structures is a proof of the quality of workmanship and empirical knowledge of the past. However, the exact reasons for such longevity and durability are difficult to determine due to their complexity.

New non-hydraulic lime based mortars are expected to have a relatively low compressive strength, approximately 0.5-3MPa (recommended by Peroni *et al.* (1981)) and are expected to adjust to seasonal and minor structural movement without damage (Gibbons TAN 1 1995, Binda *et al.* 2000). Their durability is considered rather poor, especially in conditions where the temperature drops below zero.

5.2.2. Properties and compatibility of non-hydraulic lime mortars

Sass and Snethlage (1997) defined limits within which the properties of a new repair mortar should fall to ensure its compatibility with the original one (see Chapter 3). The limits were expressed as a percentage

Figure 5.5: Historic mortar sample from Tantallon Castle, East Lothian. The paper label attached to the sample is around 3 cm across. The general texture of this internal fracture surface of an historic mortar (early 16th Century) can be seen clearly. Poorly sorted aggregate contains rounded dark basaltic rock fragments and red coloured intermediate to siliceous extrusive igneous rock fragments. There are also some more angular fragments of charcoal in this specimen.

Figure 5.6 Historic mortar sample from St Andrews Cathedral precinct wall (early 16th Century). This image shows an external weathered surface of the mortar, which is darkened compared to a fresh unweathered surface. The weathering can accentuate features like the aggregate, especially if the lime binder is preferentially weathered away.

of properties of mortars over properties of stones. Bromblet (2000) evaluated this method of compatibility determination. Mortars were made of hydrated lime and fine sand with the addition of a powder from the particular stone relevant to the repaired masonry. The mortars and stones were both tested for compressive and flexural strength, porosity, thermal and hygric dilatations, and adhesion to the surface.

The results concluded that the mortars were all porous, capillary materials with a negligible amount of soluble salts, low adhesion and a great crack sensitivity. The porosity and capillarity of the mortars were affected by the addition of the stone powder. The mortars made with stone powder were found to be closer in some properties (porosity) to the stones from which the powder was made (Bromblet 2000). In terms of compatibility with the properties of the stones only one mortar fulfilled the requirements based on the research of Sass and Snethlage (1997). It was concluded that the mortars which failed the compatibility requirements possessed too low capillarity, adhesion and mechanical strength. Or, conversely, the value of these properties in the stones was too high. The results question the appropriateness of these general compatibility limits for lime-based mortars in relation to masonry.

5.2.3. Modern and traditional renders

Marie-Victoire & Bromblet (2000) carried out a comparative experimental study on five modern cement-based mortars and three traditional lime-based mortars used for rendering in France. A number of tests was carried out including water retentivity, setting-time, shrinkage, porosity, capillarity, water and water vapour permeability, compressive and flexural strength, surface hardness and adhesion. The results confirmed that the modern ready-mixed rendering mortars are in general too impermeable and strong for conservation purposes (Marie-Victoire & Bromblet 2000). However, some of them had a number of parameters comparable with the lime-based renders which seemed to be more compatible with the masonry. Each mortar could be appropriate for different specific requirements of different masonry conservation works. This conclusion supported the use of modern materials and stressed the need for specifications of good compatibility criteria.

5.2.4. Hot mixing

Callebaut *et al.* (2000) examined properties of new non-hydraulic lime mortars in relation to a production technique known as hot mixing (sometimes also called dry mixing). Their experimental work stemmed from analysis of historic mortars and observation of a presence of white rounded lime lumps (lime inclusions) which can be associated with the hot mixing method (Leslie and Gibbons 2000). In this method quicklime is slaked while mixing with sand and water. The mortar can be applied cold, while still warm and even mixed and slaked within the structure (Gibbons 1993). Some papers indicated that these hot mixed mortars possess a better bond between the aggregate and binder as a result of etching of the surface of aggregate grains (Jedrzejewska 1967).

The results from mechanical testing showed that the hot lime mixing method produced mortars with a relatively high strength (Callebaut *et al.* 2000). Specimens were prepared from non-hydraulic lime slaked with sand and kept together for 7 days prior to casting. The values of compressive and tensile strength were compared with the results of Van Balen's (1991) earlier testing of a commercial hydrated lime mortar mix. The curing and ageing conditions were kept the same to maintain the comparability of the results. The hot mixing method produced mortars with higher mechanical strength. Callebaut *et al.* (2000) suggested that the hot mixing method had been used in the past to produce high strength and durable lime-based mortars.

Armelao *et al.* (2000) suggested that when a hot mixed mortar had been applied still hot, the higher temperature of the mortar could have favoured an interaction of calcium and silica and consequently it could have led to the creation of a better adhesion bond between these materials. The authors' objective was to study a bond between lime mortar and clay brick and they concluded that the calcium penetrates into the clay brick's pore system where it forms a layer of calcium silicate.

5.2.5. Ageing of lime putty

The effect of ageing on lime putty was studied by Hansen *et al.* (2000). The authors measured viscosity (consistency) and workability (water retention) of an aged lime putty, and flow of a mortar mix made of the aged putty. They concluded that the aged putty (16 years) performed better due to the reduction of lime particles in size with ageing. The water retention, consistency and flow tests implied that water absorbed in the older (16 years) lime putty was harder to remove by mechanical action in comparison with the younger putty (2 years). However, ageing alone may be insufficient to ensure improvement for certain types of lime. Factors such as limestone source, burning temperature, particle reactivity and slaking conditions can influence the size of crystals and affect the ageing characteristics of lime putty (Hansen *et al.*).

Thomson (2000) used a surface area test to compare particle sizes of dolomitic hydrates and putties, high calcium putty and a high calcium hydrate. The lime putty (made in laboratory) possessed very small particles. Thomson (2000) concluded that, in this case, a further reduction of the particles was very unlikely and therefore the ageing of lime putty to reduce the particles in size may not be completely relevant. According to Thomson (2000), the ageing (maturing) of lime putty provides mainly a completion of the slaking process.

5.2.6. Addition of brick dust

Addition of brick dust into lime mortars can improve their strength and durability. The results from the Smeaton project (Teutonico 1994) suggested that the clay type and its firing temperature are the factors which affect the performance of mortars. Hughes & Sugden (2000) followed this research in experimental work on hydraulic lime mortars. The authors concluded that the fineness of the brick dust and the curing conditions are the most relevant parameters to be altered in order to maximise improvement in strength. Papayianni & Theocharidou (1993) concluded that the addition of brick powder contributes to strength but it also lowers the capillary rise rate. It could also increase the water retentivity of mortars (Papayianni & Theocharidou 1993).

5.2.7. Carbonation of lime mortars

Carbonation of non-hydraulic lime mortars is considered to be the most important process of hardening and it has a direct influence on durability and strength of the mortars. Carbonation of new lime mortar can take several months, but there are also examples when carbonation of a mortar inside masonry took more than several hundred years (Hosek and Muk 1989).

Hughes *et al.* (1998) suggested that the factors that affect initial carbonation (and hardening) in the short term might have less influence on the durability and physical properties of historic mortars in the long term. Calcium carbonate is soluble and when water is present the carbonated particles can be dissolved and consequently precipitate, changing completely the pore structure and strength of a mortar. When mortar carbonates, it gains mass and its porosity decreases (Parrot 1991-92). However, in a longer term, porosity can increase due to the dissolution of calcium carbonate, mechanical deterioration, micro cracks caused by load, salt and frost attacks. As a result of this the strength and other properties of mortars vary significantly depending on ageing conditions. In Scotland there are examples of very friable mortars as well as very hard and dense mortars both made from non-hydraulic lime but exposed to different environmental conditions (Hughes 1998). Some of these relatively hard and strong mortars have a very high porosity.

During the initial hardening of mortars the rate of carbonation depends on the mortar surface finish and its permeability (Válek *et al.* 2000 Madrid). Carbonation can be slowed down by a reduction of permeability and diffusivity. Such reduction may occur due to a reduction of pore sizes induced by a progression of the carbonation inwards from the surface (Hilsdorf *et al.* no date) and inhibiting CO_2 entering deeper into the mortar.

5.3. Research into nature of mortar properties

Torraca (1988) described deterioration mechanisms of porous materials. Moisture (and its movement) is the most common degradation agent in conservation. Pore structure and various moisture transport mechanisms are therefore often correlated to durability of porous materials. A great deal of literature has been written about moisture transports in porous materials (e.g. Meng 1994). However, practical interpretation of this theoretical research is still loose. A recent example of research on salt and moisture transport in porous materials and its translation into practical suggestions for desalination of painted brick vaults has been presented by Larsen (1999). Even the properties such as porosity and/or permeability, which are commonly tested, are not well correlated to practical applications. Marie-Victoire & Bromblet (2000) pointed out the difficulties with the interpretation of porosity and permeability measurements on rendering mortars.

Understanding of the nature of properties is crucial in interpretation of the results from testing. The most significant seem to be the mechanical properties (such as compressive strength) as they relate to hardening and carbonation, and the physical properties (porosity, permeability) as they relate to durability and transport of processes within the mortars. However, the interpretation of general research for a practical application often needs additional testing and examination. One of many examples could be the research work of Papayianni & Theocharidou (1993) who tested a number of new mortar mixes in order to relate their composition, strength, absorption, absorption rate and efflorescence. The research confirmed that the pore structure is the most significant factor when describing the efflorescence tendency as it describes the moisture transport (in this case capillarity) within mortar. On the other hand, the authors concluded that open porosity and natural absorption are not good criteria for the description of moisture movement and therefore for susceptibility to efflorescence. More tests are needed to confirm these conclusions in general.

The following paragraphs inform about the latest research into properties of mortars. A great deal of papers related to material research of historic and modern mortars can also be found in RILEM conference proceedings (Bartos *et al.* 2000) about 'Historic Mortars: Characteristics and Tests'.

5.3.1. Porosity, pore structure & transport process

The amount of water in a lime mortar mix and the manner in which the water evaporates in time during setting controls the eventual porosity and the pore structure of mortars. The water content of the mix is also directly related to workability, which consequently has an influence on the quality of the compaction of mortars. Studies on cement mortars (Kroon and Crok 1961) suggested that the extent to which the mortar was compacted affected pores of 5mm in diameter and larger. During setting, as the moisture evaporates, a pore structure of the mortar is formed. The pore structure has a direct influence on permeability and other characteristics related to transport processes in the mortar. Banfill and Forster (2000) suggested a hypothetical relation between degree of hydraulicity of mortars and their gas and vapour permeability (breathability). The more hydraulic a mortar is (e.g. Portland Cement, an eminently hydraulic mortar), the less permeable it is.

Size of pores defines and limits the transport process in the porous materials as shown by Meng (1996) on studies of sandstone. For a description of the relevant porosity a correct method or combination of methods have to be used. A range of different methods and the porosity relevant to the transport processes in sandstone is shown in *Figure* 5.7 and *Figure* 5.8 taken from literature (Meng 1996). The pore structure is usually described according to pore size distribution, where the pore volume is divided into fractions corresponding to the equivalent cylindrical pore radius. It is most often measured by means of mercury intrusion porosimetry. This method allows a basic comparison of the pore structure between porous materials. It can therefore be used to characterise the most relevant transport process in a given material, i.e. a high proportion of capillary pores suggests a potential danger of capillary rise and easy transport of water. However, the porosity itself obtained by mercury intrusion does not describe the interconnection of the pores, the permeability. Moreover, only the pores which are accessible from outside are the most relevant to the weathering and the transport processes Meng (1996).

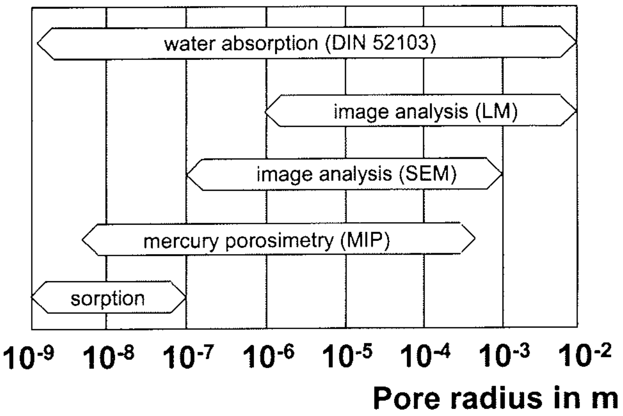


Figure 5.7: Measuring ranges of different methods for determination of porosity and pore structure (Meng 1996).

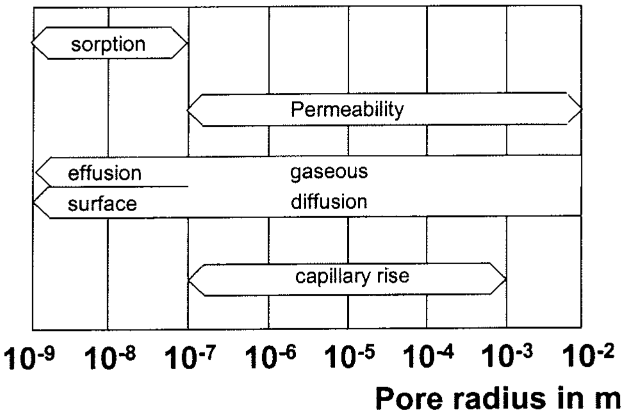
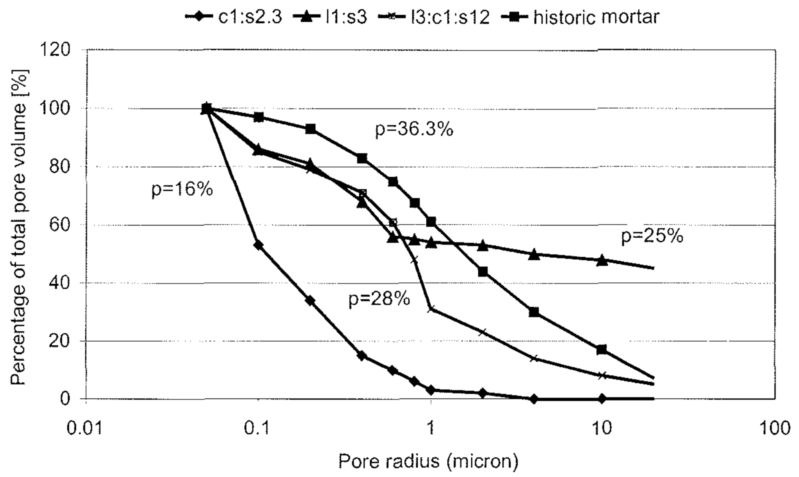


Figure 5.8: Ranges of relevant porosity to transport mechanisms (Meng 1996).

The porosity of historic mortar (from the church of Santa Marta from XV century) was measured by Biscontin *et al.* (1993) who carried out research on historic mortars in Venice. Biscontin *et al.* (1993) pointed out that all historic mortar samples analysed had similar values of pore size distribution in the range of 0.01 to 0.5mm which he related to formation during carbonation process.

Another useful characteristic of porous materials is their total porosity. It is usually measured by water absorption, a method commonly used in a building practice which covers a wide range of pores, see *Figure* 5.7 and *Figure* 5.8. Although this method may still not include exactly the whole range of pores, it measures porosity relevant to the moisture transport.



This pore size distribution graph was created from porosity measurements published in literature to explain by example the differences in pore structure of historic and modern mortars. From the graph it is obvious that the cement mortar contains a larger number of small pores than the pure lime mortar, and the cement gauged mortar lies in between them. The historic mortar had higher total porosity and fewer pores with small radius. It should be noted that the graph is only derived from a random sample presented in literature.

Figure 5.9: Comparison of cumulative pore size distributions of historic mortar (Biscontin et al. 1993) and modern mortars (Peroni et al. 1981) based on results published in literature. (p = total porosity, c-cement, s-sand, l-lime, number = proportion).

5.3.2. Carbonation

The carbonation process has been well documented. The main interest is in the carbonation of concrete in connection with corrosion of steel reinforcement (e.g. Parrot 1990). Research work on carbonation of lime-based mortars has been carried out mainly as a part of complex analysis of historic mortars. One of the few studies on new lime mortars is Van Balen's (1991 and 1994) research into modelling of lime mortar carbonation. Carbonation of non-hydraulic lime mortars is a physical-chemical process when calcium hydroxide ($Ca(OH)_2$) reacts with carbon dioxide (CO_2) from the air in presence of moisture to form calcium carbonate $CaCO_3$. This reaction is often written as a chemical equation (1):

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O \quad (1)$$

More precisely, the CO_2 diffuses from the environment into pores of mortar. According to Fick's law, the carbon dioxide travels in the pores from regions with high concentration to regions with low concentration (Papadakis *et al.* 1992). The diffusion of carbon dioxide into mortars is largely influenced by the moisture condition of the material. If the mortar is saturated, the pores are filled with moisture and the carbonation process is retarded. The diffusion of CO_2 in water is 10^4-10^5 times slower than in the air, when the pores are empty (Van Balen & Van Gemert 1994). However, a small amount of moisture has to be present in pores should the reaction happen. Carbonation is in fact a two-stage reaction.

Firstly, the carbon dioxide is dissolved in water present in pores (2a):

$$CO_2 + H_2O \leftrightarrow H_2CO_3 \quad (2a)$$

Secondly, the lime reacts with the carbonic acid to form calcium carbonate:

$$Ca(OH)_2 + H_2CO_3 \rightarrow CaCO_3 + 2H_2O \quad (2b)$$

The degree of pore saturation is therefore the main factor which controls the whole process. According to Van Balen's and Van Gemert's studies on 'Modelling lime mortar carbonation' (1994) the optimal water content for carbonation is maximum adsorption of water on surface of pores before capillary condensation. Less exact but more practical is an expression of these optimal conditions as a relative humidity of the ambient environment. Papadakis *et al.* (1992) in his paper about mathematical modelling of carbonation of concrete suggested that the maximum carbonation rate occurs at relative humidity of around 50%. Other literature recommends values between 50-60% (Hosek and Muk 1989) or 50-70% (Parrot 1991/92). These values can be compared to an average relative humidity in UK, outdoor environment, which is about 80 – 85% (Meteorological Office 1970).

The carbonation of lime mortars causes a change in their structure as well as chemistry. The formation of calcium carbonate leads to an increase in the mass and volume. This results in the gain of mass of mortar and the reduction of total porosity. Moorehead (1986) in his paper about carbonation of hydrated lime explains that the mass gain is about 35% of hydrated lime used, which may convert to an 11.8% increase of volume. The increase of volume is internal, inside the pore structure of the mortar, without any significant effect on the overall alteration. These changes of pore structure and the pore size distribution were reported in studies on concrete (Hilsdorf *et al.* 1995, Pihlajavaara 1968). In concrete, carbonation causes an increase of capillary porosity in the range of 0.0075mm to 0.100μm (Hilsdorf *et al.* 1995). For a hydrated lime mortar a graph of pore size distribution which showed a change in pore diameters was explained in the literature (Moorehead 1986). It described a reduction of pore sizes around 1mm and larger, as well as an increase of pores of a smaller size within a range of 0.05μm to 0.5μm, see *Figure 5.10* . However, the range of the pore sizes affected by carbonation depended on the initial porosity and the pore size distribution existing prior to carbonation.

The external factors are:

- Relative humidity (moisture conditions), drying and wetting cycles, and wind speed.
- Temperature.
  Solubility of carbon dioxide decreases with increasing temperature. However, reactivity is better with higher temperature. The optimal temperature is therefore about 20°C (Van Balen & Van Gemert 1994).
- Content of carbon dioxide in the ambient environment.
  Content of CO_2 in the air is normally around 0.03% (Hosek and Muk 1989).

The internal factors are:

- Porosity and permeability of the material.
  This governs transport of moisture, and diffusion of carbon dioxide (porosity and permeability depend on the composition of the mortar mix).
- Composition, quality of lime and quality of slaking, etc.

Table 5.3: Summary of the factors influencing the carbonation process divided into external and internal ones.

A reduction of pore sizes induced by carbonation results in reduction of permeability and diffusivity of mortars (Hilsdorf *et al.* 1995). This consequently leads to the following conditions:

(i) Diffusion of carbon dioxide becomes more difficult with depth and therefore carbonation in deeper parts of mortar is slower; in the case of combination with other conditions which are unfavourable for carbonation, it can be retarded or stopped altogether.

(ii) Reduction of porosity and permeability should enhance durability of mortars as was pointed out in literature (Hilsdorf *et al.* 1995). However, this may in general be a positive effect for concrete, but mortars with high permeability are sometimes required in conservation works.

The increase of mass due to carbonation can be used as an indirect measurement of the carbonation progress. Parrot (1991-92), following the work of others (Kamimura *et al.* 1965), examined an increase of mass of concrete due to carbonation. In his experimental work he concluded that the mass gains are directly related to gains of carbon dioxide as presented in *Figure 5.11* . From this relationship it was suggested that the kinetics of carbonation could be assessed indirectly by monitoring the increase of mass in time.

'Secondary' Carbonation and Other Reactions

Carbonation reaction results in the formation of calcium carbonate, which is chemically relatively stable. This has a positive effect overall on the durability of mortars. During the carbonation process, the pH value of 12.5 of uncarbonated calcium hydroxide is reduced to a pH value of around 8.4.

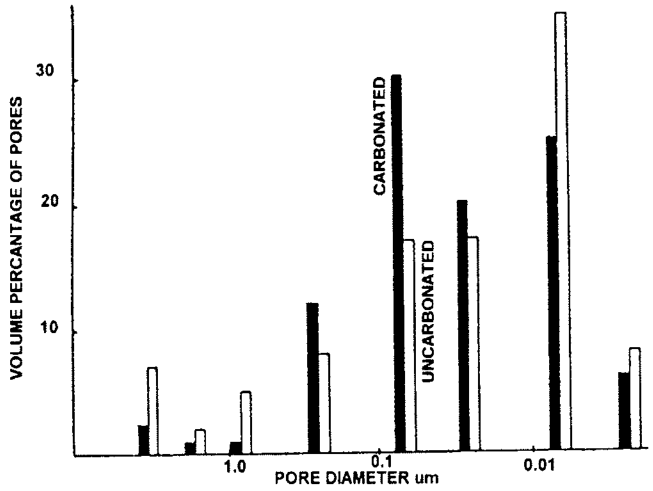


Figure 5.10: Change in pore size distribution of hydrated lime compact according to Moorehead's studies (1986).

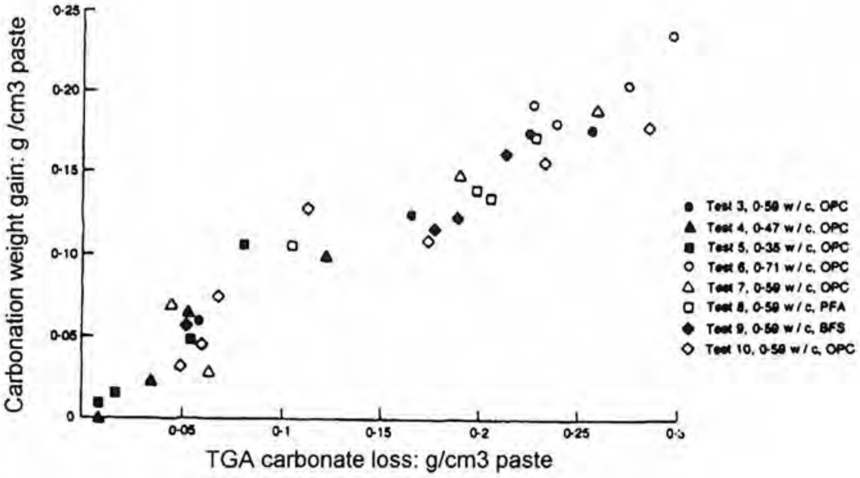


Figure 5.11: Mass gains due to carbonation against calcium carbonate loss from thermogravimetric analysis, according to Parrot (1991-92).

However, calcium carbonate is not an entirely insoluble material and further chemical reactions, which affect the durability of lime mortars, can occur. This can be a process of dissolution of Ca, which is described by a chemical equation (3):

$$CaCO_3 + H_2CO_3 \rightarrow Ca(HCO_3)_2 \quad (3)$$

In this process calcium carbonate reacts with carbonic acid to form calcium bicarbonate. A higher solubility of the calcium bicarbonate leads to a gradual dissolution of Ca, which often means enlargement of pores and a gradual decrease of the strength and durability of the mortar (Cowie and Glasser 1991-92). In building practice, this process is called lime leaching. It requires the presence of water and therefore, once a mortar is carbonated, water should not be allowed to flow through. Lime leaching can be denominated according to the place where the precipitation of calcium carbonate occurs, which mainly depends on the water transport mechanisms.

- **Internally.** It can precipitate inside the mortar pores, cavities, cracks, between layers of two mortars or in the interface of mortar and masonry. The existence of this process should be considered when assessing long term durability of the mortars. In some cases the carbonates precipitate in cracks, commonly called re-healing, which can be seen even as a positive and durability enhancing feature. To some extent the dissolution of the calcium carbonate occurs in all lime-based mortars. This is sometimes called two-stage carbonation, where the second stage is the dissolution of calcium carbonate and its recrystallisation, which brings about an increase in the size of crystals and binds them more tightly together. It gives greater tightness and strength to the mortars (Chandra 1998).

Internal lime leaching has been observed on many historic mortars, e.g. historic harling from Gylen Castle, see *Figure 5.12*.

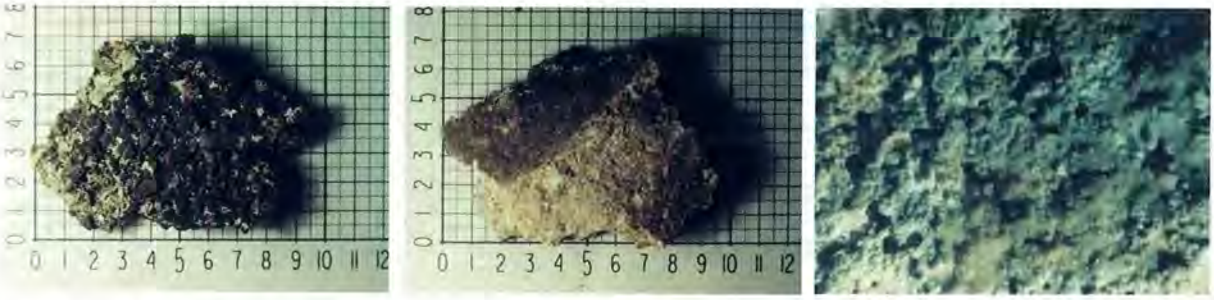


Figure 5.12: Example of internal lime leaching on the interface between lime mortar harling and stone masonry from Gylen Castle. From left: external side of harling (scale in cm), internal side of harling– internal lime leaching (scale in cm), detail of internal lime leaching (scale: 7.3mm across the picture).

- **Externally**. The dissolved carbonate is transported by water out from the mortar, where it precipitates on the external masonry or render surface. It can be caused by unidirectional flow of water through the mortar. From a conservation point of view, external lime leaching is highly undesirable. It not only weakens the mortar but also leaves hard lime staining on the masonry face, see *Figure 5.13*.

A parallel phenomenon to lime leaching is the transport of uncarbonated lime in the mortar. For example, very rapid drying can draw the lime particles (calcium hydroxide) to the surface and make the surface whiter (Gibbons TAN 1). Too high water content in the fresh mortar mix can have the same effect (Gibbons TAN 1). An example of such whitening of the surface is presented in *Figure 5.13*

Dissolved calcium carbonate and uncarbonated calcium hydroxide can be transported by water and both are available for further reactions. The recrystallisation process of carbonate particles within the mortar is a common process. Perander and Råman (1985) pointed out that the conditions required for this type of repeated carbonation consist of open pore structure (mortar's surface), prolonged period of time and moist air. The air however, has to be free of airborne pollutants, namely free of sulphur dioxide. Sulphur dioxide is the most aggressive towards mortars containing lime (Sabbioni *et al.* 2001). However, these conditions – air free of atmospheric pollution - cannot be fulfilled today in urban areas. Zappia *et al.* (1994) described the reaction between airborne sulphur compounds and mortars. In general, it leads to the formation of calcium sulphate, which crystallises as gypsum and subsequently forms a black crust commonly observed on limestone monuments. The pattern of such soiling is determined by the construction and detailing of the masonry. Calcium sulphate can be washed away by rainwater but it precipitates in sheltered areas. In the case of cement mortars and hydraulic mortars, the gypsum and the hydraulic constituents can interact to form other salts such as ettringite and thaumasite (Zappia *et al.* 1994). The formation of gypsum, ettringite and thaumasite is accompanied by a series of physico-chemical processes such as decomposition of binder, volume expansion and cracking, which brings about a loss of cohesion in the mortars (Sabbioni *et al.* 2001).

5.3.3. Strength

Strength development and testing of cements and other hydraulic compounds have been explained elsewhere (Neville 1963, Hewlett 1998).

According to Hosek and Muk (1989) the strength of non-hydraulic lime mortars is gained from three processes.

Figure 5.13: Doune Castle; external lime leaching on the left hand side and new lime repointing, slightly pink colour is becoming whiter by the uncarbonated lime transported on the surface on the right hand side.

- Drying of lime mortars.
- A long-term dissolution of silica in the alkaline environment of calcium hydroxide and consequent formation of solid phases of calcium silicate.
- Carbonation of lime mortars.

In general, carbonation is considered to have the biggest influence on strength development. However, this requires deeper studies, as the main influence on strength seems to differ depending on the ageing stages and the curing conditions. The initial stage of hardening seems to be more influenced by the drying out process than carbonation (Válek and Bartos 2001).

Schäfer & Hilsdorf (1993) tried to relate type of binder, binder/aggregate proportion and porosity in order to indirectly estimate compressive strength and elastic modulus of historic mortars. The authors introduced a formula to calculate the strength purely from results of chemical and visual analysis.

Suter and Song (1995) pointed out that when describing strength of historic mortars it is important to perform a relevant test rather than rely on the wide range of data presented in publications. A great variety of mortars with various proportions of binder and aggregate have been reported in literature (Suter and Song 1995). However, the size and number of samples available from historic masonry restricts the mechanical testing. Often non-destructive, 'in-situ' testing is necessary for testing mortars within historic masonry.

Testing standard specimens of a new mortar may not correspond to the characteristics of mortars from within masonry. Henzel and Karl (1987) demonstrated that strength obtained from standard laboratory specimens was lower than that obtained from a normal mortar joint. The mortar from within masonry was subjected to various influential factors which affected its properties. One of the differences is the contrast between mortar prepared in a steel mould and mortar in masonry units with certain moisture conditions and suction.

5.3.4. Bond between mortar and masonry

The bond between mortar and masonry is important for both stress-strain transmission and durability. If the bond is poor, water can penetrate much more easily inside masonry and cause degradation. This applies to bedding mortar but also to plasters and renders.

The bond between mortar and masonry unit has been studied for a number of years. The investigations are mainly into Portland cement based mortars and bricks, and there seem to be few direct applications of these research results for traditional lime and stone masonry. The literature review by Goodwin & West (1980) show that the quality of the bond is supposed to be influenced mainly by mortar (composition, consistency and water retentivity), bricks (absorption and texture) and workmanship. A good quality bond binds units well together providing structurally sound and watertight masonry. The quality of the bond should not be measured solely by the adhesive strength. It was indicated that conditions which provide the high strength bond may not be the same as those which provide resistance to water penetration of the bond (Goodwin & West 1980). Durability and quality of mortars is affected by the suction of the bricks. High suction can lead to a shortage of water when hydration takes place and affect the bond. On the other hand, Harrison (1986) in his experimental work demonstrated that high suction bricks improved the durability of cement based mortars compared with specimens cast within 'zero' suction bricks.

The most important single factor affecting the bond was concluded to be the absorption rate of masonry units (Goodwin & West 1980). Groot (1993) described the effects of water on mortar/brick bond by measuring water content changes and flow rates immediately after bricklaying using a neutron transmission technique. He concluded that no relation could be established between the water content at the interface and the bond strength. The flow rate was more critical. For the tested specimens, the most significant differences in flow rates occurred during the first 100-200 seconds after bricklaying. Transport of fine particles of binder towards the interface was observed. Interestingly, neither the transport nor the bond was affected by pre-wetting. The type of brick influenced the flow rate much more than aggregate grading. Groot (1995) later on suggested a model for water transport between mortar and brick immediately after bricklaying. The brick mortar model was described by means of a hydraulic diameter model (brick) and a particle capillary pressure theory (mortar). Such a theory can be used for the design of a 'compatible' mortar-brick combination.

It is possible that lime can penetrate into bricks and carbonate in their pores, therefore creating a physical adhesion between the mortar and the brick (Armelao *et al.* 2000). Similar observation was presented earlier by Baronio and Binda (1987) who studied a mortar-brick interface on samples of historic and modern mortars. The authors noticed that the extent and conditions of a lime-based mortar/brick contact was better than the one of a cement-based mortar as the lime penetrates to the open porosity of the bricks. Cement based mortars presented long narrow voids on the contact with bricks.

5.4. Testing of mortars

Any new mortar should be tested prior to its application for various reasons. In conservation, testing can be seen as part of a complex logical procedure to ensure correct selection and application of the mortar (see Table, Charola *et al.* 1997). The range of the tests varies depending on the mortar's functions and applications (described by Sass and Snethlage 1997). For example, performance characteristics of mortars should be tested to ensure their compatibility with historic materials.

Most tests currently available for evaluation of properties of mortars are standardised. However, these tests are not always applicable for conservation purposes. In the case of lime mortars especially, the testing requires a better understanding of their performance and their correct application. A typical example is the standardised testing of compressive strength of non-hydraulic lime mortars, which requires completely different specimen preparation and curing than cement-based mortars for which the standards are written. As a consequence of the lack of appropriate tests the standard tests have to be modified and new tests designed to be applicable in conservation. This problem is well known and discussed (Sickels-Taves 1999, ASTM congress on 'The Use of and Need for Preservation Standards in Architectural Conservation'). It is generally recognised that one of the main values of standard testing is the ability to compare the results on an international level. However, the comparison of the results is not always straightforward, as there are a number of national standards with different testing methods (Henriques and Charola 1996). In conservation, the aim of standard testing is to avoid the introduction of potentially harmful treatment (Teutonico *et al.* 1997, Dahlem workshop). New standard methods for testing compatibility of mortars are still under development. However, there have been a number of suggestions and first attempts which are currently under discussion.

- Objective of the treatment (e.g. consolidation, repointing)
- Requisites of the treatment (specifications e.g. matching the original mortar in colour and texture, new mortar should be less strong than the original mortar and masonry)
- Selection of the relevant physico-chemical parameters needed to evaluate the effects of the treatment and of the experimental methods to measure above parameters.
- Selection of suitable samples (e.g. type, size)
- Selection of application method and its implementation (e.g. correct workmanship, standardised preparation of specimens – cubes, prisms)
- Selection of weathering conditions (e.g. natural, standardised, accelerated)
- Comparison and critical evaluation of the results obtained on treated and untreated samples, and before and after ageing (compatibility, comparison of parameters of historic and new mortars, carbonation of non-hydraulic limes is a slow process - the age factor is very important)

Table 5.4: The steps of a testing protocol to follow suggested by Charola et al. (1997) with examples given for repointing of masonry.

The new standard testing of lime mortars should consider two main aspects (Charola & Henriques 1999):

- How to prepare, treat and test the specimens
- What kind of tests should be carried out to characterise the mortars

One of the main problems of laboratory testing is that it does not conform to the behaviour of mortar in masonry. The specimens should be prepared according to the appropriate practice. Tests, such as compressive strength, should incorporate the slow nature of the hardening process. The size of the specimen can influence the results. For example, the smaller a specimen of non-hydraulic lime mortar is, the faster it is completely carbonated (Baronio *et al.* 2000).

The other aspect considers the appropriate tests to be carried out in order to characterise mortars. A selection of tests is available (e.g. Peroni *et al.* 1981, Rossi-Doria 1986), however the aim should be to define a minimum number of tests sufficient to characterise the mortar (Charola & Henriques 1999). For compatibility compliance, a range of optimal performance within which the results should fall must be resolved (Charola & Henriques 1999).

Time of setting
Compressive resistance
Modulus of elasticity
Adherence strength
Thermal and hygroscopic expansion
Release of soluble salts
Capillary water absorption
Water vapour permeability
24-h immersion water absorption (for brick masonry)
Resistance to chlorides and sulphates

Table 5.5: Proposed tests for standard testing of lime based mortars (open for discussion) by Charola & Henriques (1999).

A definition of the exact number of tests and the range of optimal results expected from the mortars is, however, difficult since the conservation tasks are unique, the historic buildings are unique and they require individual solutions (Burman 1997). The selection of tests should be based on a holistic acquaintance with all relevant information incorporating the uniqueness of historic structure.

At the moment it can be confirmed that the testing itself can be standardised as there has been a number of tests completed on mortars. This should improve the descriptions of the properties related to the function of the mortars in real structures. However, what tests should be carried out, and the limits for the compatibility requirements which would provide the selection of the right repair mortar, still need further studies.

5.4.1. Examples of experimental studies and testing of mortars

Research projects dealing with the experimental study and testing of new mortars often developed from a practical design of a new repair mortar (e.g. The Smeaton Project, Teutonico *et al.* 1994, Perander and Råman 1985, Fontaine 1999). These projects set up certain standards and often serve as examples for similar projects in the future. Some of them are reviewed in the following paragraphs.

(i) English Heritage, ICCROM and Bournemouth University joint research project (Teutonico *et al.* 1994) started as an identification of a suitable mortar for repair of Hadrian's Wall. Three types of mortars were examined (Lime: Sand: Brick dust; Lime: Sand: Cement; Lime: Sand: Brick dust: Cement). Pure lime: sand mortar was not examined. The mortars were prepared to the same workability by an English Heritage craftsman and were cast into wooden moulds. The specimens were cured at 25°C and 90% RH for 120 days. Depth of carbonation was determined by phenolphthalein pH solution with the maximum depth 17mm (fully carbonated mortar). The compressive strength was tested on 100mm cubes and, prior to testing, the samples were immersed in water for 24 hours. Other tests, such as moisture content, stiffening ratio, water vapour permeability and sodium sulphate crystallisation were carried out. This briefly summarises phase 1 of the project, which led to the following conclusions:

- Addition of brick dust alters the properties of lime mortars.
- The low-fired brick dust seems to have the most positive effect on durability and strength of the mortars.
- Addition of a small quantity of cement has a negative effect on the strength and durability of the mortars. (Note that in literature similar conclusions were reached (Holmström (1995), however, opposite ones also exist e.g. Von Konow (1998).)

Further research investigated pozzolanic additives. It identified the positive influence of brick dust on strength and durability of mortars. The effect of the addition of brick dust later on became the objective of a consequent research. The results indicated that brick dust with a lower size particle range (range <75 microns) could act as reactive pozzolanas. Also, a low-fired brick dust (temperature < 900°C) seemed to have the most positive effect on the strength and durability of lime: sand: brick dust mix (Teutonico *et al.* 2000).

Based on the Smeaton project a new experimental research was initiated into hydraulic limes and their blend with different proportions of non-hydraulic lime (Teutonico *et al.* 2000). The preliminary results suggested that water vapour permeability appears to be lower with hydraulicity. The addition of a significant proportion of lime putty into hydraulic lime:sand mortar mix significantly reduces its compressive strength and durability performance from a salt crystallisation test.

The Smeaton Project evaluated the mortar specimens according to compressive strength, which was assumed to be the appropriate measure of their performance and related to their durability. The mortar mixes used were not designed to be compatible with any particular properties of the original mortar or masonry. Instead, general specifications were used. A significant number of various mortar mixes was tested and similar tests were used for all mortar mixes. As such it offered a good deal of comparison between different mortars. The main contribution of these projects may be seen in the formulation of testing procedures and obtaining background information about properties of different mortar mixes.

(ii) Perander and Råman (1985) published a research report about mortars in Finland which, apart from general information and analysis of historic mortars, comprised also a design of a new repair mortar. The authors described the variability of historic lime mortars including different burning, slaking and mixing. However, they concluded that the differences between the lime slaking had no significant influence on the properties of lime mortars examined in the laboratory. Moreover, they found the non-hydraulic lime-based mortars unsuitable for exterior renders in the Finnish climate. Their recommendation for repair mortar was based on composition of 60% of lime and the remaining 40% of some hydraulic additives (cement, fly ash, trass etc.). The recommended ratio of binder:aggregate was 1:4 – 1:5.

In their research on development of new repair mortar the authors focused on the influence of hydraulic and air-entraining additives. A number of standard tests was carried out to compare different mortar mixes (including consistency and stiffening, tensile and compressive strength, water absorption and porosity, frost resistance etc.). However, the development of a repair mortar did not comprise any requirements of compatibility with historic mortars and the evaluation parameters were unclear. Determination of what the desired universal repair mortar was supposed to be was lacking. Although this research cannot be considered as a guide for design and direct selection of a repair mortar, it offered fundamental results for comparison between lime mortars with different hydraulic additives and described various methods of testing.

(iii) Fontaine *et al.* (1999) presented an overview of testing repair mortars. The study was based on experimental work with repair mortars and their practical application in the Canadian environment (Suter *et al.* 1995). The authors highlighted problems with the testing of repair mortars, as there were few standard tests directly applicable to them and virtually no standard test that would assess their durability. Modifications to incorporate the conservation view of some current standards designed for modern buildings were suggested and discussed. The authors pointed out that every testing should be supplemented with specification criteria which should be fulfilled. Special focus was given to the frost resistance of mortars and their mechanical properties. Certain characteristics such as, for example, 'breathing' of mortars were considered important but no tests had been developed to describe the relevant properties.

From practical experience of testing repair mortars and in-situ assessment of their performance certain performance criteria were recommended to ensure compatibility (see Table 5.6). The results offered certain practical implications for mixing and composition of mortars. Pure and hydraulic lime mortars did not meet the performance (Table 5.6) criteria defined, mainly due to their poor frost resistance. Only the mortars gauged with cement were able to comply with these criteria. This was found to be in accordance with the findings from in-situ assessments, that lime mortars typically disintegrated within 5 to 10 years after their application.

Performance characteristics	**Limits**	**Explanatory remarks**
Compressive strength of mortar	1 to 8 MPa	Compromise between too strong and dense mortar and too weak, which cracks and allows water ingress. Bedding mortar should be a minimum of 2MPa.
Split tensile/ compressive strength of mortar	≥ 10%	Low tensile strength results in cracking of mortars. This could be considered as a material quality measure for brittle materials, which have a ratio between compressive and tensile strength of around 10. The tensile strength rarely exceeds the bond strength for cement and lime mortars therefore there is no upper limit defined.
Young's modulus of mortar	1 to 8Gpa	Describes deformability of mortars under stress. Mortars are valued for ability to adjust to a minor movement. Too stiff mortar can cause cracking to the adjacent material. However, this depends also on the elastic (Young's) moduli of all materials involved.
Flexural bond of masonry	≥ 0.3MPa	Ideally the interface (the bond) of mortar and stone should be as strong as the mortar.
Expansion (freeze/thaw test) of masonry	≤ 0.04%	Unidirectional freeze/thaw test where the damage is quantified by the change in the width of the mortar joint. Expansion between 0.04 to 0.4% is considered marginal.

Table 5.6: Performance criteria for new pointing mortars according to Fontaine et al. (1999) with remarks to the tests' applicability and their limits.

Fontaine *et al.* (1999) described testing and selection criteria for repair mortars with respect to the Canadian environment and therefore an emphasis was on durability (freeze/thaw). It recognised the need for compatibility and conservation related criteria for repair mortars in general. The values limiting the required performance were created from realistic expectations of the material properties, practical experience with repair and research results. All considerations were put together, resulting in a limitation of the properties. However, two aspects should be highlighted which were missing in the research discussed above.

- The appropriateness of the limits related to compatibility should have been verified by testing the compatibility of both materials together. The compatibility is about two materials - not just the mortar.
- Exact limits may be applicable just for certain conditions e.g. Canadian environment. Traditional masonry in Canada is different from, for example, that of Europe, but certain differences may exist even on a smaller scale. Such variability should be considered when a new standard test is proposed.

The overview of these three projects demonstrated the current situation in the search for a better repair mortar. Current standard tests were found to be not relevant, as they did not reflect any conservation requirements. The selection criteria for mortars could be based on certain performance tests, but the relevance of these tests to the compatibility or conservation requirements is yet to be confirmed. Typically, when a design of new mortar was described it began with compatibility 'introduction' but the results and conclusions were only about the tests themselves, not about the ultimate objective – the design and selection of the compatible repair mortar. In general, comparative testing of mortar specimens in a laboratory always leads to the selection of some mortar but there is no proof of how this relates to real conditions and compatibility. It should be remembered that the mortar selected from a certain limited number of specimens is only the best mortar available from the limited number of specimens, not necessarily the most appropriate for the repair.

On the other hand, the practical approach to the design and testing of repair mortars is very effective as it narrows the number of variables to the most realistic ones. It means that each material has its certain range of achievable properties and use. Non-hydraulic lime mortar cannot be expected to possess a high compressive strength or good freeze/thaw durability. Through experimental testing and practical experience it is possible to clarify these limits and use each material in its proper way.

5.4.2. Factors affecting the preparation of specimens and testing mortars

The performance of modern non-hydraulic lime mortars made of lime putty is considered to be strongly dependent on workmanship (Gibbons 1995 TAN 1). The workmanship comprises not only the application of mortar but also particular constructional details, adequate workability, final surface finish, in-situ protection and curing. The workmanship should reflect the actual state of masonry and environmental conditions. Also, the moisture suction parameters of masonry units together with ageing conditions affect the bond between mortar and masonry.

Water

It has been suggested by Schäfer *et al.* (1993) that a correlation between porosity of an ancient and new lime mortar could be used as a method to estimate the compressive strength. The amount of water added determines porosity of a hardened mortar. The water in non-hydraulic lime mortars can evaporate or be absorbed by the adjacent masonry. Together with a degree of compaction they characterise a volume of voids in the mortar. Higher porosity means lower strength. The mechanical properties of lime mortars are improved if the amount of water is reduced (Torraca 1988). The same applies for cement-based or hydraulic lime mortars, however, a certain amount of water is needed for the hydraulic reaction (Neville 1963). Lime putty usually contains enough water for mortar (bedding and/or pointing mortar) to be prepared without a further addition of water (TAN 1 1995).

Aggregate

From research into concrete it is known that the influence of aggregate on strength of mortar is due not only to the mechanical strength of aggregate but also, to a considerable degree, to its absorption and bond characteristics (Neville 1963). Papayianni *et al.* (1993 Paris) suggested that historic mortars in Greece follow the same principles of aggregate proportioning as those which are valid for concrete. However, the grading of aggregate of historic mortars does not comply with current standards (it contains a considerable amount of fine argillaceous components and a relatively high proportion of course grains (Schäfer & Hilsdorf 1993)). The strength of a mortar depends on the strength of a weaker component in the mortar mix. In the case of lime based mortar, it is usually the lime matrix which determines the overall strength. Moreover, the shape of the aggregate and its surface control the mechanical bond between binder and aggregate. Type of sand influences the mechanical properties of hardened lime mortars (Callebaut *et al.* 2000). Aggregate grading also affects workability and consequently shrinkage of mortars. Sánchez (1997) summarised that aggregate, its maximum size, grading and proportion of the finest particles affect shrinkage of mortars. Mixtures of fine and coarse aggregate are considered the best, as such mixtures occupy space in the most efficient way (Torraca 1995).

Casting Specimens into Moulds

The contact surface of moulds with the specimens, suction and entrance of air influences mainly lime-based mortars (Charola & Henriques 1999). Therefore, properties of lime mortar made in a steel mould are different from those of mortar cast between masonry (Lawrence & Samarasinghe, 2000). They depend mainly on the mould surface, the size of the specimen and how long the specimen was left in the mould. Mortar specimens for investigation of properties should be prepared in a realistic way. Lawrence & Samarasinghe (2000) suggested producing and curing mortar specimens between two masonry units.

Workability

Type of aggregate, its grading, type of lime (age of lime putty) and lime/aggregate proportions control the amount of water needed to provide good workability. The optimal water/binder proportions differ depending on construction and application techniques. Good compaction of lime mortar is vital for its performance. Good workable lime mortar possesses a greater degree of plasticity; it is often described as similar to a modelling clay.

Mixing

The mixing and production methods of lime mortars can also have a very strong influence on their performance. Maturing of some lime putties reduces their particle size and improves their water retentivity (Hansen *et al.* 2000). The sand carrying capacity of lime mortar should improve with reduction of particle size (Gibbons 1995 TAN 1). Hand and different mechanical mixing can produced mortars of different quality. From practice it is known that the mortar plasticity can be improved by the method of mixing. Traditional techniques of mixing 'by hand' involved beating, chopping and ramming on a wooden board until the mix was sticky and workable (Gibbons 1995 TAN 1). Ready-mixed mortar should be re-mixed before use.

In relation to the performance of mortars assessed by standard testing, Henriques & Charola (1996) assessed the effect of mixing and preparation on compressive strength of pozzolanic lime mortars. The two standard procedures prescribed by British Standard (BS 4551) and European specifications (EN 196-1) resulted in pozzolanic mortar specimens with different mechanical properties. The pure lime mortar specimens, however, did not show any significant differences in their mechanical properties for these two different preparation and mixing standards. The mechanical properties of both non-hydraulic and pozzolanic mortars were also affected by different curing conditions prescribed by British Standard (BS 4551) and French specifications (CSTB). Wetter conditions (BS 4551) favoured hydration but slowed down carbonation (Henriques & Charola (1996).

Curing

The curing/ageing conditions for non-hydraulic lime mortars promote a combination of drying out and carbonation at such a rate that minimises shrinkage. The ideal environment to achieve a maximum rate of carbonation, has a temperature around 20ºC and relative humidity between 50-70% (Van Balen & Van Gemert 1994). The strength development of lime-based mortars due to carbonation is inherently a very long-term process, depending on the curing/ageing conditions.

Curing described by British Standard (BS 4551: Part 1: 1998) for mortar specimens is not appropriate for non-hydraulic lime mortars. It assumes the presence of a hydraulic binder, which requires a damp environment or immersion to allow hydration of the hydraulic components. Moist curing in a container can possibly be used, however, the container should not be airtight. According to literature (Parrot 1991-1992) and considering the nature of the hardening process of lime mortar, such humid conditions can retard carbonation and do not represent ambient conditions encountered in practice.

The common 28 days' curing is not sufficient for non-hydraulic lime mortars (Charola and Henriques 1999). A length of curing to provide a comparable strength testing can be difficult to predict. It depends on the curing conditions, moulding and size of the sample. Accelerated curing where the specimens were exposed to a higher CO_2 concentration was described (Knöfel and Schubert 1993). This accelerated curing was developed to make testing of lime mortars available at 28 days. The effects of the specimen size and the length of curing on the strength testing of lime mortars have been observed in many research papers (e.g. Baronio *et al.* 2000).

5.4.3. Fresh mortars

Mortars, while fresh, are tested in order to ensure a certain standard quality. The tests are described in detail in national standards (e.g. British Standard BS 4551: Part 1 Methods of testing mortars, screeds and plasters) EN 1015 Methods of tests for Mortar for Masonry 1999. Of these tests, the workability test is the most beneficial for practice. It combines needs for appropriate consistency of the material regarding its application but also its properties after setting and hardening.

Workability Testing

Recent research into properties of mortars applied various methods to measure workability, as the standardised methods are not always suitable for mortar mixes used in conservation. The Smeaton project (Teutonico *et al.* 1994) employed an English Heritage master craftsman to judge the suitable consistency to produce comparable specimens of mortar. In the experimental work carried out by Papayianni *et al.* (1993) a flow table test (British Standard BS 4551) was used to achieve comparable consistency and workability between various mortar mixes. The water contents of mortars were adjusted to achieve the same flow value (170 ± 1mm). Sickels (1987) in her PhD research used a British Standard test, *Determination of Consistency by Dropping Ball*, to provide the same consistency between mortars.

Schäfer *et al.* (1993) did not measure the workability directly at all, but instead applied a theory of water demand for mortar. To achieve the same consistency, the water demand was calculated from water/binder ratio considering that different types of binder have different water requirements. The calculation was based on sizes of the binder particles. The smaller particles have higher surface area and therefore require more water in order to achieve the same consistency. However, the consistency and water demand based on particle sizes may not be fully relevant to the workability, especially not between binders of a different nature. Hansen *et al.* (2000) in their research into ageing of lime putty demonstrated that the older lime with smaller particles and lower water content showed more plasticity and water retentively than the younger one (with larger particles). To achieve the same consistency, more water was needed for the lime with smaller particles. For the workability of lime, however, mortar characteristics such as plasticity and water retentivity were more important.

British Standard BS 4551: Part 1 describes two tests which are used to assess the workability of mortars - *Determination of Consistency by Dropping Ball* and *Determination of Flow* (flow table). It should be recognised that these tests are designed for 'modern' mortars and modern construction methods and therefore they are not fully applicable for assessment of lime based mortars used in conservation. Válek (2000) in his PhD thesis concluded that the consistency measurement by dropping ball is not applicable for lime-mortars made of lime putty. However, he recommended the flow table combined with determination of moisture content for a laboratory measurement of workability. On the other hand, Callebaut (2000, PhD thesis) pointed out that for practical use the flow table needs more research as the measured values vary depending on the mortar mix. Callebaut *et al.* (2000) also considered the standardised flow table test to be unsuitable for determination of consistency of different lime mortars. To a degree, for lime-based mortars the suitability and unsuitability of the flow table was explained by Hansen *et al.* (2000) in their research into ageing of lime putty. The authors suggested that for some lime mortars the flow test is less sensitive than others to the water content of fresh mortars. Older lime putty with smaller particles (larger surface area) was less sensitive for variation of water content than younger lime putty. The flow table may not be precise enough to compare workability of mortars with different binders or even with the same kind of binder but with different properties.

An alternative to the flow table in the measurement of workability of conservation mortars could be the standard plasticity test (ASTM C110) using the Emley plasticimeter. It is an American test which is not commonly used in Europe. Thomson (2000) used the Emley test in her comparative study of dolomitic and high-calcium lime mortars. She pointed out that this test incorporates two important workability characteristics (water retention and spreadability of mortars), hence the test relates to the workability of mortars.

The subjective judgement of workability by a skilled craftsman should not be underestimated. It is an important factor which influences the quality of mortar; in fact, in practice the craftsman alone controls the amount of added water. The Smeaton Project (Teutonico *et al.* 1994) confirmed the precision of such judgement (English Heritage master craftsman) by measuring moisture contents of the specimens. The moisture content ranged from 15 to 19%, which was considered to be relatively consistent.

5.4.4. Hardened mortars

Properties of hardened mortars, as well as fresh mortars, should ideally be determined by means of standard testing methods. However, these standard testing methods often need to be modified to suit peculiarities of conservation practice. The following paragraphs deal with both standard and modified tests of hardened mortars with the objective of describing these tests within the context of their use. There are therefore described tests which were used recently in research and experimental projects, and also in practical designs of composition of new mortar and/or compatibility testing. The selection of the tests and testing procedures reviewed depended on available literature – it is not a compete list of testing methods applicable for mortars.

Shrinkage

The shrinkage of mortar is its volume reduction caused by changes of temperature due to a hydration process or to a loss of water through evaporation. Contraction caused by shrinkage generates internal stresses and, when restricted, it can result in cracks and a failure of mortar. Cracks can develop at any time before, during and after hardening when a mortar is not able to deform (Sánchez *et al.* 1997). Shrinkage is a complex phenomenon depending on many factors incorporating material properties, drying and hardening conditions. Faster shrinkage (for example mortar cured in fast drying conditions) produces a greater danger of cracks (Sánchez *et al.* 1997).

Sánchez *et al.* (1997) tested shrinkage of lime based mortars on specimens according to standards for cement based mortars. The authors considered only shrinkage caused by evaporation (hydraulic shrinkage). Shrinkage due to carbonation was considered negligible because of its slow course. The authors compared shrinkage of lime mortars made with two sands, standard sand and sand found in historic mortars, during the first 28 days (starting at the day 1). The main influential factors on the shrinkage of the mortars were considered to be the maximum size of aggregate and its compactness, water/lime ratio, content of fines, and strain module of aggregate. Testing confirmed the influence of some of these factors. The shrinkage (volume reduction) took place mainly during the first day. However, no cracks developed as the mortar was still in a relatively plastic state. The shrinkage was measured in a range from 1.15 to 1.6%. Application of the methodology from cement mortars to measure the shrinkage of lime mortars was successful.

Mechanical Strength-Strain Testing

Strength is an important property of mortars. In a design of a new mortar it should be a key parameter for the assessment of compatibility between mortars. However, unlike in modern industry, in conservation it should not be used as a sole measure of quality. In fact, for the determination of compatibility of non-hydraulic lime mortars the compressive strength alone may not be needed at all. Nevertheless, in a research into mechanical and physical properties of lime mortars, compressive strength testing is a standard way of assessing their hardening, setting and strength, and is related to carbonation, porosity and other physical properties (Válek 2001).

Baronio *et al.* (2000) described experimental research into testing of mortars prepared according to the properties of historic mortars. The study comprised analysis of historic mortar, production of new mortar and its curing followed by experimental testing on non-hydraulic lime mortar specimens made of putty and hydrate. The authors tested the development of compressive and flexural strength on specimens of different dimensions and preparation, and cured under different conditions. Comparison of the specimens of different sizes showed that the smaller ones (20x20x120mm) acquired maximum strength faster than the larger specimens (40x40x160mm). On the other hand, the results obtained from the larger specimens were more consistent and less scattered. The authors suggested that the larger specimens (70x70mm) possessed lower strength than the smaller specimens due to the size of aggregate. The proportion between dimensions of specimens and the maximum size particles of aggregate was considered to affect the strength. Additionally, the open porosity of the mortars was measured, however, no correlation between open porosity and compressive strength has been drawn.

Válek & Bartos (2001) examined the effect of curing conditions on strength development of non-hydraulic lime mortar specimens. The strongest specimens were obtained from dry indoor conditions, where they also acquired strength faster than the ones cured outdoors. The drying out process appeared to be more significant for strength development in the early stages (first six months) than the degree of carbonation. Less variable curing conditions resulted in less variable strength. The authors also concluded that the addition of water increased the porosity and decreased the strength of non-hydraulic lime mortars. However, there was a certain margin within which the effect of added water on porosity and strength was not clearly recognisable and only a larger amount of water added resulted in the changes of porosity and strength.

Binda *et al.* (2000) studied mortar of thick jointed Byzantine masonry. Masonry prisms were built for laboratory trials as a reproduction of a historic masonry based on a complex analysis of mortar composition and in-situ testing. The prisms were used to study mechanical behaviour (deformation) of the mortar and bricks under compression and its development with ageing of the specimens. Deformation in the mortar joints improved with age but, as expected, it remained higher than the deformation of the bricks. Stiffening of the 'wall' increased with age although its strength was not much affected by ageing. The authors concluded that the thick joints of Byzantine masonry allowed large deformation as hardening of mortar occurred slowly. For example, therefore, a soil settlement at early stages did not cause any significant cracks of the masonry construction. The high deformability of mortar also allowed withstanding of tensile stress caused by compression of bricks but without diminishing the overall strength of the masonry. This research confirmed one of the hypotheses about a degree of deformability of lime mortars, which are often implied to possess high deformability and the ability to eliminate or reduce the local stresses in the masonry joints (Schäfer & Hilsdorf 1993).

Karaveziroglou & Papayianni (1993) studied the stress capacity of masonry with thick mortar joints. In order to carry out the testing, masonry prisms were built using modern bricks manufactured to match the properties (density, modulus of elasticity and compressive strength) of the historic ones. Four different mortar compositions were used. All of them contained lime, pozzolana, brick dust and sand, and two mixes were 'enhanced' with cement. Bedding joints were 20 and 40mm thick. The compressive strength of masonry prisms increased with the compressive strength of mortar. The masonry with thicker joints had lower compressive strength than that with the 20mm joints. The compressive strength obtained was higher than usually assumed and the authors pointed out that the compressive strength of old masonry with thick joints might previously have been under estimated.

Adhesion/Bond

Budelmann (1993) concluded that in order to achieve a high adhesion bond between mortar and masonry a mortar mix based on hydraulic binder is needed. The author carried out tests on masonry units joined with mortars. Such masonry couples were mechanically tested in tension. Apart from the hydraulic binder, the adhesive strength was determined by some properties of the stone such as open porosity, pore size distribution and capillarity suction (Budelmann 1993).

Marie-Victoire & Bromblet (2000) tested adhesion of modern and traditional renders by a pull out test. The outcome consisted of two results: the maximum load and the location of the failure. The test method therefore conveniently combined two main parameters. Maximum load described a quality of the bond and in general a higher load was desirable. The other parameter, a location of failure, was a 'compatibility' limitation which determined whether or not the particular adhesion of the mortar could potentially cause any damage to the masonry when pulled away. The results confirmed that for traditional lime-based mortars (hydraulic and putty mortar) the failure occurred within the thickness of render. For the modern ready-mixed renders the location of failure varied depending on the substrate masonry and probably on the quality of workmanship during application. The modern ready mixed renders performed quite well in terms of adhesion, however, the wrong type of failure occurred occasionally. The authors stated clearly that such material would be unacceptable for conservation of historic buildings.

Other simple adhesion tests can be carried out for a basic evaluation. Perander and Råman (1985) used a simple test for testing renders on bricks. Rendered brick was stroked from the back with a hammer and then the number of samples where mortar remained attached to the brick was counted.

Carbonation

The most common way to determine the depth of carbonation of lime or cement based mortars is by a Phenolphthalein pH indicator (a method recommended by the RILEM committee (RILEM draft recommendation 1984)). The carbonation process reduces the pH value of 12.5 of Calcium hydroxide ($Ca(OH)_2$) to pH values below 9 of Calcium Carbonate ($CaCO_3$). When the sample is sprayed with the Phenolphthalein pH indicator it changes its colour from colourless to magenta in a region where the pH value is higher than 9. An advantage of this method is that it offers a non-destructive and very efficient measurement. However, this method does not recognise a partially carbonated mortar - only fully carbonated mortar can be determined (Ohgishi *et al.* 1993). Therefore, the method cannot be used to evaluate quantitatively carbonation of lime mortars (Válek 2000). In addition, Parrot *et al.* (1989), who measured a gradient of neutralisation zone on 36-year-old concrete members, pointed out that a certain penetration zone should be considered when describing the carbonation depth. The detailed carbonation gradient was determined by thermogravimetric analysis (TGA) of samples taken by drilling at 5mm depth intervals (Parrot *et al.* 1989). Especially when a mortar has aged in indoor conditions, the partially carbonated zone can be more than 20 or 30mm wide (Parrot *et al.* 1989).

A good indication for laboratory observation of the carbonation progress of mortar specimens is a change of weight (Parrot 1991-92). Parrot (1991-92) in his experimental work on concrete, following findings of Kamimura *et al.* (1965), concluded that the mass gains of mortar are directly related to the gains of carbon dioxide. From this relationship it was suggested that the kinetics of carbonation could be assessed indirectly by monitoring an increase of weight in time. This method of measurement of carbonation was applied on lime mortar specimens by Válek (2000).

Porosity and Pore Structure

Measurement of porosity by water absorption is a method commonly used in building practice. The method measures open porosity in total and it covers a wide range of pores. The porosity of mortars is determined by immersing the specimens in water under vacuum for a certain length of time. The mass of water absorbed in a specimen is the difference between the oven dried mass (at 105 ± 5°C) and the vacuum saturated mass. More precisely, porosity is defined as a percentage of a volume of a mortar's pore space in a total volume of mortar. The porosity measurement is accompanied by a 24 hours' water saturation coefficient. It is a measure of the extent to which the pores are filled when the mortar is exposed and allowed to absorb water. The sample was immersed in water for 24 hours. The average value of this coefficient was 0.71. These simple methods are recommended for assessment of durability of building stones (Ross & Butlin 1989). Other national standards use this method but its procedure varies mainly in the length of time specimens are left to saturate in vacuum and naturally.

As discussed earlier, the amount of mixing water, however, strongly influences the initial pore structure of the mortar, its compaction and application. Researching the reproduction of historic mortars, Hoffmann *et al.* (1993) varied proportions of binder, aggregate and water to identify their influence on the porosity and pore size distribution. Increasing the binder proportion in mortar increased the amount of finer pores. Decreasing the grain size of the sand fractions caused an increase of porosity and water absorption coefficient. The authors pointed out that the reproducibility (measured by pore size distribution) of mortars is very difficult even though the same preparation process of specimens is followed. Samples of historic mortars can be used to determine composition, porosity and pore size distribution, however, there is no correlation to the original content of mixing water (Hoffmann *et al.* 1993). Microporosity of lime mortars can correlate to their frost sensitivity but perhaps less to capillary transport (Hoffmann *et al.* 1993). Unfortunately, the authors did not describe the degree of carbonation of the measured specimens. From the discussion on porosity and carbonation it seems that the microporosity depends on the level of carbonation progress. Many micropores can be closed with carbonation in progress and the whole pore spectrum can be expected to change with the reduction of the pores.

Harrison (1986), experimenting with durability of cement/lime mortars, measured the porosity of different mortar mixes. For each binder six different mixes, incorporating six different sand gradings, were prepared to possess the same consistency. The finer the sand was, the more water was required to achieve the required consistency. This resulted in higher porosity of mortars with finer sands. The specimens were made as small brick prisms to simulate realistically the suction of real masonry. The pore size distribution of mortars was affected by the suction of bricks within which the mortar was laid. Mortars cured within high suction bricks seemed to have a slightly higher proportion of larger pores. Interesting was the method of measurement of the porosity which combined the mercury porosimetry (pores between 0.01μm to 3μm) and estimation of larger pores under optical microscope (pores from 45μm to 1mm). Results of these two methods added together were checked by the total porosity.

A similar experimental procedure was repeated later by Harrison and Gaze (1990). The authors noted that the cement mortars with air-entraining agents produced a mixture of spherical closed pores (formed by the air-entraining agents) and continuous pore system formed by water. The cement mortars gauged with lime contained a continuous pore system only. Both mixes were prepared to the same consistency and had a similar level of porosity.

Research by Perander and Råman (1985) on the influence of air-entraining additives to mortars showed that the water absorption under normal atmospheric pressure was not greatly affected by the additives. However, in underpressure the effect of air-entraining additives is more pronounced. This is applicable mainly for mortar mixes containing cement.

Permeability

When the pores are interconnected it allows the passage of a fluid usually described by permeability. Permeability is a flow property of a porous medium which is strictly related to the flow that occurs under an applied pressure differential. It is frequently used to cover other transport mechanisms such as absorption and diffusion. Depending on the fluid, different kinds of permeability can be measured. The most common ones are water, water vapour, and gas permeability.

Gas permeability especially is currently seen as a suitable method for characterisation of porous materials in relation to their durability. Válek (2000) measured gas permeability of lime mortars and masonry materials and pointed out that it is a novel method for non-destructive in-situ testing of masonry materials. The potential of the technique was later confirmed by Curran & Smith (2000), who compared changes of gas permeability of a sandstone block in an urban area. Válek *et al.* (2000) compared differences between gas permeability of cleaned and uncleaned sandstone blocks. A comparative study of the relation between total open porosity, carbonation and permeability of lime mortar specimens was carried out by Válek et al. (2000, Euromat 99). The gas permeability of mortar specimens varied depending on the surface finish. The surface finish and its gas permeability affected the carbonation more than the porosity.

Carey and Curran (2000) described the creation of a contoured permeability map of a sandstone block. The authors (Válek (2000, Carey and Curran (2000)) all agree that there is a great potential for the use of gas permeability measurements, however, there is still need for more research to verify the results and correlate them to a direct application in conservation.

5.4.5. Durability of mortars

The most common causes of degradation are frost and sulphate attack. Both cause internal mechanical deterioration through an expansion in the pore structure of mortars. Therefore, the mortars and masonry materials are tested for liability to frost and salt attacks. The causes and effects of weathering in porous materials used in buildings have been widely described in literature (Torraca 1988). Grimm (1985) reviewed literature on the durability of brick masonry. In practice the durability of masonry can be summarised as its resistance to water penetration (Charola & Lazzarini 1986). Cracks and measured compressive strength did not show any correlation to the durability of historic mortars (Von Konow 1993).

In the laboratory, the durability of mortars can be tested on specimens subjected to accelerated weather conditions. The procedure of these tests, number of cycles etc., can vary depending on the tests but recently there have been a number of attempts to standardise these procedures (e.g. RILEM recommendations.) The dilemma of this kind of testing is in its relevance to real masonry constructions. Mortar within masonry is usually only unidirectionally exposed to the agents and causes of deterioration. Moreover, mortar within the masonry interacts with the masonry in terms of moisture distribution and transport processes, which are affected by their pore structure. Consequently, the pore structure of mortars is formed by this interaction, plus conditions during the hardening process and ageing process. Lime-based mortars are not one hundred percent chemically stable and a redistribution of lime binder can occur. On the other hand, laboratory testing represents a good comparable method for selection of the most durable material under general conditions.

Another testing of durability can utilise an exposure site, which represents usually harsher but more realistic conditions. Such sites offer full-scale testing and offer the most realistic testing conditions. However, full-scale testing on exposure sites has it is own limitations in terms of practicality, cost efficiency, comparability of the results, repeatability of the experiments and time required for exposure.

Harrison (1986) tested the durability of cement-based mortars in relation to the properties affected by composition, mixing and curing. The specimens were cast and cured within a couple of dry and wet bricks representing high and zero suction respectively. At the age of 28 days, the specimens were exposed to accelerated weathering by freeze/thaw, wetting and drying with sulphates and a combination of both sulphates and freeze/thaw cycles. This testing procedure was repeated later on, incorporating more cement mortar mixes (Harrison and Gaze 1990). Both papers came to similar conclusions. It seemed that durability and strength improved by curing the mortar within dry bricks. This was attributed to the suction of the bricks, which removed a certain amount of water from the mortars immediately after the completion of the masonry units. Air-entraining additives and dry bricks (mortar was dried by suction of dry bricks) affected the porosity and improved the frost resistance of mortars. A higher amount of cement produced higher strength and mortars with higher cement content were more resistant to frost. On the other hand, mortars containing lime (and sulphate resisting Portland cement) were better able to resist sulphate attack. The combination of sulphate and frost attack was the most severe test. Gradation of sand affected the mixing, workability and water content, which consequently affected the porosity and strength. However, the papers did not come to any general conclusion relating strength, porosity and durability apart from the above practical observation.

Waldum & Anda (2000) studied degradation of lime render sample panels. The authors pointed out general implications that non-hydraulic lime mortars do not have a good reputation in terms of their durability in freeze/thaw and salt deterioration. From their measurement the authors observed that the beginning of the degradation process correlates with an increase of the concentration of Ca in run-off water. They did not observe any salt degradation as the freeze/thaw was too dominating.

The Smeaton Project (Teutonico 1994) concluded that small quantities of cement in mortar mixes with cement content less than 1:3:12 (cement:lime:sand) had a negative effect on their strength and durability. A similar conclusion was reached by Holmström (1995) who stated that the durability of lime mortars gauged with cement decreased, even in comparison to lime-based mortars, if the proportion of added cement is less than 40-50% in weight to the lime. This may be related to salt deterioration, as even small quantities of cement increase the salinity of mortars (Papayianni & Theocharidou 1993).

Maurenbrecher *et al.* (2000) tested small stack bonded stone masonry specimens to examine the freeze/thaw durability of lime mortars gauged with cement. The mortars consisted of 1:2:8 (cement:lime:sand) and were tested after 28 days of curing. Masonry prisms were exposed to unidirectional freezing. Loss of bond was found to be the most common failure as nearly all mortars showed a bond failure after 60 cycles. All mortars containing lime putty failed. It was noted that the stone properties affected the mortars. The mortar which did not fail the test was the mortar with the higher proportion of cement (1:1:6). However, no comparison was made with pure lime mortars.

Von Konow's (1998) work on the influence of aggregate grading on the performance of various mortar mixes used for repair pointed out a correlation between grain size of aggregate which affects the capillary suction and therefore the frost resistance. She concluded that a greater proportion of filler than normally recommended gives the mortar homogeneous and dense packing. This, together with a higher proportion of a finer aggregate, improved the frost resistance.

A new, interesting method for testing durability of mortars for abrasive damage was presented by Lawrence & Samarasinghe (2000). The authors tried to relate abrasive durability to surface hardness, not the strength or hardness of mortar in general. They suggested salt cycling test for laboratory testing and scratch test for in-situ testing. A salt (5% solution of sodium chloride) was used in an accelerated test to crystallise at the surface to cause mechanical degradation. The scratch test involved repeated scratching of a mortar surface by a probe with a constant force. The correlation between these two tests suggested that both tests measure the same property.

5.4.6. 'In-situ' testing of properties of mortars

'In-situ' testing and determination of mortar properties is desirable and in conservation is often the only way of their determination due to sampling restriction. Ideally, the testing methods should be non-destructive. For determination of mechanical properties of mortars and masonry a number of new methods are emerging but few of them are proven to be able to replace actual laboratory testing. 'In-situ', non-destructive testing measures other physical characteristics, which correlate to the actual tested property. It is therefore obvious that the quality of the results often depends on the quality of the correlation. However, 'in-situ' testing offers one advantage over laboratory testing: it can determine the value of compressive strength, permeability, etc., under real conditions.

To determine mechanical properties of mortars or concretes core samples are usually taken and tested in the laboratory. The core samples required are too large (BS 1881 on testing concrete cores recommends min. 100mm in diameter) to be drilled from historic masonry. A new research into minimising the core diameter is dependent on the size of aggregate. For example, for aggregate with a maximum size of particles of 30mm in diameter, microcores with diameter of 28mm can be drilled (Indelicato 1993).

The compressive strength of mortars can also be determined from correlation of the strength and energy needed to drill a small cavity. The method suggested by Gucci & Barsotti (1995) seems to be particularly applicable to mortars with lower compressive strength than 4MPa, in which case it offers a good value regardless of the mechanical properties of the sand. Although the damage is minimal, and in both cases the structural soundness and stability is not affected, the methods are not non-destructive.

Hendry (1993) suggested that for the calculation of strength of historic masonry where lime mortar was used, and no direct tests can be carried out, a nominal 1.0 N/mm^2 may be taken. Non-destructive testing can be used to assess uniformity and the presence of hidden defects in masonry (Hendry 1993).

The hardness of mortars and their strength is usually assessed by subjective methods involving scraping and scratching. A more objective way of assessment was described by Van Der Klugt (1991), who suggested a classification of the quality of pointing mortars based on their hardness. The hardness was measured by modified Schmidt pendulum hammer where the bigger is the recoil of the pendulum hammer, the harder is the surface. The method is applicable to bedding mortar (measuring head is 5mm in diameter) or plasters.

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