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Understanding the lime cycle and its influence on historical construction practice

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Burning, slaking and carbonation are the major steps in the lime-cycle leading to the air-hardening of lime mortar. Lime mortar has been used since antiquity. Its preparation and its use have been understood by generations although during time schemes of understanding have changed.

Looking today at historical perceptions of the lime cycle helps to identify the proper understanding of certain material properties and the use of lime mortar in the past.

First the lime cycle is explained based on actual chemistry and mineralogy. Looking at some well known writings of Vitruvius, Renaissance authors, C. Perrault, 18th century authors, the evolution of the understanding of the role of heat, water, air in the preparation and the use of lime becomes clear.

Impurities in the limestone selected for burning can be responsible for hydraulic reactions in lime considered too often as being only air-hardening. Interpreting sources as Vitruvius and later ones we could deduce that such stones have been selected to prepare lime. This demonstrates that also hydraulic hardening is to be considered beside carbonation if some constrains are respected. This understanding throws a new insight on what is hydraulic lime and could contribute to a better use and standardisation of building lime, even today.

Some ways to prepare lime (e.g. dry slaking) have been investigated in laboratory conditions. Lime mortars with a similar appearance as historic mortar samples could be produced. Dry slaking solves a number of problems met today in using building lime and is compatible with the use of hydraulic lime if impurities were present in the burned limestone. It seems obvious that different types of lime could have been used for different applications with lime.

The objective of this paper is to explain thoroughly the use of lime as a building material and to relate different types of lime to certain types of application in construction history as we can know from actual scientific knowledge confronted with some historical sources.

Introduction

This paper aims to clarify different ways to prepare lime and to explain in which way this preparation influenced the use of lime over time. The understanding of the «lime cycle» and the hardening of lime and hydraulic lime over time is the subject of this contribution. Recent research has contributed to a better understanding and therefore this information will be used to update information that, although known to some, was not available to a wider scientific audience.[2] This paper will concentrate on the North-Western experiences and context although for its «origins» of understand we have to start in the Mediterranean Basin.

So far one of the most important and often referred to source of information on the use of lime in mortars in antiquity is Vitruvius. Most of us today refer to translations of this document. Progress in understanding the properties of materials have certainly also induced some new interpretations and translations of Vitruvius «Ten books of architecture» (Vitruvius [25 B.C.], 1914). In the case of the sources of hydraulic binders new interpretations have been proposed by F. Davidovits (1994) and also by others based on a better integration of natural with human sciences.

Our contribution begins in the Roman period acknowledging however that lime mortar was used earlier by the Greeks mainly for rendering. They used sand that sometimes was from volcanic origins as explained by Roland Martin (1965), amongst others. Mortars for rendering also contained plaster and marble powder. The use of earth of Santorini and crushed ceramics showed that Greek were able to increase hydraulicity of the mortar artificially. The product made in this manner is the predecessor of the artificial pozzolana (Furlan and Bissegger 1975).

ROMAN LIME MORTAR.

Romans developed and utilized the technology of lime mortar and lime concrete. They even used different aggregates to control the density of the concrete with lime in a way to assure the stability of domes (Lamprecht 1986).

The extent of the Roman Empire is at the origin of the wide dissemination of this knowledge in Europe and around the Mediterranean Sea.

One of the oldest descriptions of «opus caementicum» can be found in the writings of Cato the Elder (234–149 B.C.) (Van Tyghem 1966). He described a construction «ex calce et caementis». Vitruvius gives the most accurate description of the composition and the use of lime mortar. In his book «Ten books on architecture», probably a good description of the building practice at that time (Adam 1984, ref. 113), different data are given on the technology of lime mortars at that time (Vitruvius [25 B.C.] 1914, 45).

> After slaking it (lime), mix your mortar, if using pit sand, in the proportions three parts of sand to one of lime; if using river or sea-sand, mix two parts of sand to one of lime . . . , in using river or sea sand, the addition of a third part composed of burnt brick, pounded up and sifted, will make your mortar of a better composition to use.» (Vitruvius [25 B.C.] 1914, II, V, 1)

Although we concentrate on the lime cycle (burning - slaking - carbonation) it should not be forgotten that the use of inert fillers (as sand) and reactive fillers (as pozzolanic materials) were understood to change the properties of lime mortar. Sea-sand seems is a lesser quality sand to use according to Vitruvius because there is the danger for efflorescence.

Although they had no proper —in our terms— explanation for it, the Romans new that adding earth from Puzzuoli (and other places like the hills of Mysia in the west of Turkey, in the surroundings of Mount Etna) give mortar hydraulic properties.

The Romans made the link between the volcanic origin of the earth and the property and they tried using their philosophy of nature to give an explanation.

Vitruvius suggested using this pozzolana to make the mortar resistant against water.

Although the Romans knew natural pozzolana, it seems that they were not able to choose the right limestone to make natural hydraulic lime. It has been shown that Romans in Great Britain made hydraulic lime with high calcium and artificial pozzolana as powdered tiles instead of using the clayish limestone in the direct environment with which it should have been possible to make natural hydraulic lime (Davey 1961, 104).

Pliny the Younger, Seneca and Sidonius Apolinaris also wrote about the effect of the use of natural pozzolana on lime mortar (Ferrari 1968).

Many legends exist about the secrets of the composition of good Roman mortars and about the use of additives as egg white, casein and oil. Probably those additives add to lime mortar, were reserved for particular applications. Oil has been used in the mortars for the sealing of ceramic water pipes (Malinowski 1979, Malinowski 1981, Malinowski 1982) and also Vitruvius described the use of oil for the sealing of the joints of watertight floors. (Vitruvius [25 B.C.] 1914, VII, 1.7.)

Raw materials and treatment.

Apart from the composition the choice of the raw material and the treatment were also of great importance to the quality of the Roman mortar (Frizot 1977).

When lime mortar was used for massive parts of masonry and floors the lime mortar was well rammed whereby its density increased. The execution of rendering in different layers with different composition and the polishing at the end influenced the water transport in the mortar and also the carbonation process (Malinowski 1979, Malinowski 1981, Malinowski 1982). All evidence seems to indicate that the good quality of the Roman mortars was due to a good control of the firing process of limestone, the homogeneity of the mortar (concrete) and the skilful application.

Burning of lime

J.P Adam (Adam 1984, 69–90) distinguishes three types of burning in the Mediterranean region in antiquity:

1. The first is the burning in a kiln in which the fire is made at the bottom and the kiln is filled with limestone lumps. The firing is discontinue as the burned stones have to be taken out at once from the whole kiln, this is an intermittent type of kiln
2. The second way is burning in a kiln filled with alternating layers of limestone and fuel. Burned limestone is taken out at the bottom while new layers of limestone and fuel are brought in at the top. The firing is continued, as the firing does not have to stop when the quicklime is taken out; this is a continuous type of kiln
3. The third method is the firing in open air. This method can only be used at lower temperature as for gypsum stone to prepare plaster.[3]

Cato the Elder prescribes the construction of a kiln, dug into in a mountain slope so as to avoid cooling by the wind. The homogeneity of the temperature in the kiln at about 900°C was very important for the quality of the lime (Adam 1984; Van Balen 1991).

In some regions with much rain, the top of the oven was closed to avoid fast cooling and some circumferential holes were used for the evacuation of the smoke and to fill the kiln. We don't know whether such kilns were used in the North-West of Europe at that time. We do know that more recent kilns in the region of Tournai had an opening at the top by which it could be filled. From the Roman period, the archaeological remains of one Roman kiln have been found in Tournai (Chantry 1979).

The slaking

Slaking often occurred at the unloading dockyard, as the transport of quicklime was less heavy. Smaller quantities were transported in amphora at sites where it was not possible to slack lime (Adam 1984, p.78, fig. 160) and to keep it for a certain time.

Slaking has to guarantee that all quicklime has turned to calcium hydroxide. Therefore the lime had to lie in pits for a certain time before being used it in mortar; Pliny prescribed 3 years (Adam 1984, ref.108). For the same reason a good mixing and knocking up of sand and lime is important. This was done with a lime chopper (the Roman *ascia*) that was able to crush lumps in the quick lime.

There is a strong prejudice that considers that this is the only way slaking occurred. More and more research is filling the gap that existed in our knowledge on the use of high calcium lime versus hydraulic lime. This allows us thus to consider a variety of ways of slaking and preparing lime and lime mortar.

Roman understanding of the lime cycle

The term «lime cycle» is generally used to describe the sequence of processes leading from limestone ($CaCO_3$) to Carbonated lime ($CaCO_3$) being again of the same chemical nature. Intermediate processes are the *burning* changing $CaCO_3$ to CaO (quicklime) followed by the *slaking* changing CaO into $Ca(OH)_2$ ((slaked)lime or portlandite) and followed by the *carbonation* or hardening changing into $Ca(OH)_2$ into $CaCO_3$.

In his second book, chapter V, §2 and §3, Vitruvius explains the lime cycle. He describes how lime mortar hardens and how lime can be used to make mortar:

> The reason why lime makes a solid structure on being combined with water and sand seems to be this: that rocks, like all other bodies, are composed of the four elements. Those which contain a large proportion of air are soft; of water, are tough from the moisture; of earth, hard; and of fire, more brittle.
>
> Therefore, if limestone, without being burned, is merely pounded up small and then mixed with sand and so put into the work, the mass does not solidify nor can it hold together. But if the stone is thrown into the kiln, it loses its former property of solidity by exposure to the great heat of the fire, and so with its strength burnt out and exhausted it is left with its pores open and empty. Hence, the moisture and air in the body of the stone being burned out and set free, and only a residuum of heat being left, lying in it, if the stone is then immersed in water, the moisture, before the water can feel the influence of the fire, makes its way into the open pores; then the stone begins to get hot, and finally, after it cools off, the heat is rejected from the body of the lime.
>
> Consequently, limestone when taken out of the kiln cannot be as heavy as when it was thrown in, but on being weighed, though its bulk remains the same, it is found to have lost about a third of its weight owing to boiling out of the water. Therefore, its pores being thus opened and its texture rendered loose, it readily mixes with sand, and hence the two materials cohere as they dry, unite with the rubble, and make a solid structure.

According to Vitruvius the burning is necessary to open the structure by which it can glue together to the other materials in the mortar.

Effect of pozzolana.

In his book II, chapter 6 Vitruvius wrote about the mixture of lime and volcanic sand to make a mortar resistant to water and even able to harden under water. He gave the following explanation:

> the soils on the slopes of the mountains in these neighbourhoods (where pozzolana are found) is hot and full of hot springs. This would not be so unless the mountains had beneath them huge fires of burning sulphur or alum or asphalt. So the fire and the heat of the flames, coming up from far within through the fissures, make the soil there light, and the tufa found there is spongy and free from moisture. Hence, when the three substances (lime, pozzolana, tufa), all formed on a similar principle by the force of fire, are mixed together, the water suddenly taken in makes them cohere, and the moisture which neither the waves nor the forces of the water can dissolve.

Ancient authors stressed using pure limestone to guarantee a good quality of lime.

The quality was controlled by weighing the loss of substance with burning. This allowed some scientists to conclude that this certainly must have been the prime reason of the late discovery of natural hydraulic lime (Alou 1989, p.5). Romans have been said to make their hydraulic mortar by adding natural (pozzolanic earth) or artificial (crushed tiles) pozzolanic additives rather than to use nearby sources of limestone able to produce hydraulic lime (Davey 1961).

We should however have questions about these conclusions for a number of reasons

1. According to Vitruvius a good limestone for producing lime should lose about one third of its weight (Vitruvius [25 BC] 1914, book II, Chapter V, §3]. One third being much less than the stochiometric 44% of weight loss for a pure limestone, considering even the stone to be completely dry. This difference in weight loss can be accepted if either some (hydraulic) impurity in the limestone are present or if some of the limestone hasn't been burned enough to be completely converted into quicklime.
2. Another reason could also be that the time span between the slaking and the use of the lime for the mortar is having an influence on how appropriate a lime is considered for mortars. As long as mortar is slaked and used within a week (up to 2 weeks at the maximum) the hydraulic lime component is still contributing to the binding and is not considered as a deficit in the binder. Calcination or burning a clayish limestone up to 900°C will result in the making of â-C_2S that takes a long time to set completely (Bertoldi, 1987)(Callebaut et al. 2000).
3. A third reason to doubt, is related to the previous one; if a part of the hydraulic fraction of the lime becomes an inert filler due to premature setting, the mortar composition can be adapted in the mixing faze to compensate this lack of (active) binder.

The above arguments are valid for lime mortar (for masonry) and are not necessary valid for the use of lime for fine renderings or for lime paints which might require other rheological properties.

LIME TECHNOLOGY IN MEDIEVAL TIME

It might be considered a long step in time to go from Roman period to the medieval but from the period in between no much progress on (scientific) understanding on the use of lime can be reported. In our regions different illustrations and texts of that period have contributed to a better understanding of the way lime and lime mortar was used. For the illustrations we will refer to the publication in which they can be found instead of representing them here.

The technology of making lime did not considerably change until the eighteenth —nineteenth century. The poorly organised building industry and the supply of raw materials obliged the builders to look for their building materials in their immediate environment. This explains the variety of compositions in the mortars found through the chemical analysis of ancient mortars nowadays. Also the hydraulicity index[4] of the mortars analysed varies considerably. This index seems to be the most discriminative element in the comparison of different mortars analysed by chemical methods. This index reflects something of the practice in a way that the type of limestone and/or the use of artificial or natural pozzolana can be traced back.

A natural pozzolana used at that time in Belgium, the Netherlands and Germany was and still is named trass. Trass is a powdered tuff stone from the Rhine Valley; its quality depends on the composition of the raw material (N. 1967). As in the Roman period artificial pozzolana were mainly crushed ceramics (tiles, bricks, . . .). Natural hydraulic lime was available on the borders of the river Scheldt in the neighbourhood of Tournai. The stone of these geological layers has been used in Romanesque and early Gothic period almost everywhere it could be transported. Probably also the same limestone was transported for making lime. Recent research on a limited set of mortar samples of civil architecture in Tournai seems to indicate that, however close to the mentioned quarries, no hydraulic lime was used for masonry mortar (Callebaut 2000).

Composition of lime mortars based on chemical analyses.

Different analytical methods are being used for the identification of the composition of lime mortars. The following publications give relevant recent information on those techniques: (Van Balen e.a. 2000; Callebaut 2000; Callebaut e.a. 2001; Ellis 1999; Hughes and Cuthbert. 2000; Hughes e.a. 2001; Lindquist and Sandström. 2000; Martinet and Quenee. 1999; Mueller and Hansen. 2001; Prado e.a. 2001; Radonjic e.a. 2001; Schlütter e.a. 2001; Winnefeld and Knöfel. 2001).

Chemical analysis can help to compare mortars with each other and help to identify different building periods. It is the first method that has been used to analyse mortars with the intention to date them. In the beginning when Jedrezewska (Jedrzejewska 1960; Jedrzejewska 1967; Jedrzejewska 1981) started the research on chemical analyses it was hoped that this method would give an absolute date of mortar samples by identifying the evolution of the mortar composition with time. Today this is considered a unrealistic endeavour.

Chemical analysis can give some information on the composition of the mortar, although the exact composition can only be found when the raw material (lime, sand, . . .) are available. The dependence on the local available material makes this research very difficult.

The study of the grain size distribution of the sand can be helpful in identifying mortars, as it can help to identify the sand used for preparing the mortar.[5]

From the analyses of different mortars for rendering and masonry, Wisser (Wisser and Knöfel, 1988) found that the sand to lime ratio was higher then in Roman times. They found an average of 28.5 weight percentage lime in the mortars. Many other analyses seem to confirm this high lime content in our regions. Analyses carried out at the Laboratorium Reyntjens showed that the lime content of different samples from the Cathedral of Antwerp varied from 16% to 55%[6] (Van Balen and Van Gemert. 1990).

Due to the high amount of lime the question arose whether such a lime mortar could have been used without having problems with shrinkage (Wisser and Knöfel, 1988). The authors' conclusion was that they must have been slaking the lime with (wet) sand, which explains also the presence of the typical lime piths in the mortar. Slaking with sand necessitated less water and the shrinkage afterwards must have decreased.[7]

The dissertation of K. Callebaut exactly studied this assumptions and he has demonstrated that the slaking of lime with sand is feasible and corresponds with the findings in ancient mortars. He also showed that this slaking technique results in a mortar with an acceptable higher binder content and a lower water demand (Callebaut 2000, 181)

Lime production and sale

Some aspects related to the production of lime mortar are illustrated in the works of F. Van Tyghem (Van Tyghem 1966), Binding and Nussbaum (1978) and Du Colombier (1953). They studied the medieval building practice through iconographical representations.

Lime production

During the Middle Ages two types of kilns were used: continuously and intermittent working kilns. In the intermittent kiln it took 3 to 4 days to heat the kiln and to allow it to cool enough to take out the burned limestone. This way of working corresponded very much with the description given by Cato. The continuously working kilns were used in the same way as in antiquity.

Before the sixteenth century very few limekilns had been depicted on paintings or drawings. More representations are known from the sixteenth century on. One of the oldest representation of a lime kiln can be found in a Sicilian mosaic from the 12th century in the Capella Palatina in Palermo (Van Tyghem 1966).

Limekilns were either built beside the river that was used to transport the limestone, as was the case for the Flanders' kiln represented on an engraving of the townscape seen from the other side of the Scheldt.[8] In the kiln at the border of the Scheldt in Antwerp probably limestone of the Tournai region must have been burned.

Limekilns were also built beside the great building sites or at places where limestone and/or fuel was available. Until the end of XIII° wood was primarily used as fuel. To burn lime, an important amount of wood, coal or peat was needed.[9] Due to a better energy balance the amount of coals in a continue kiln could be diminished with 20% (Davey 1961).

To produce 1 ton of lime the quantity of wood necessary corresponded with an oak of 46 cm in diameter and a length of 9 m, or two pine trees with the same dimensions. It is obvious that the consumption of such amounts of timber, not only for lime burning but also for brick firing caused problems of shortage[10] and therefore other sources were sought. More and more coal was used but this fuel produced significant air pollution. Therefore new regulations were issued at the beginning of the 14th century e.g. in London (Salzman refered to in (Davey 1961) to reduce the impact of air pollution on the population.

For the same reason the magistrate of Brussels issued some measures in 1415–1416, and in 1536 it was forbidden in Amsterdam to make or set up a limekiln within one mile of the town (Van De Walle 1959, 65).

In the Netherlands mainly seashells were used for producing lime, as there is almost no limestone source available, this was also the case in the 14th century. As a source of energy often peat was used in the lime kilns (Janse 1981).

Burning lime was a job for skilled workmen and the work continued over night as the kiln had to burn continuously. Therefore the lime-burners were paid well (Salzman 1965, 152). Carrying limestone and the lime was a job for unskilled workmen:

> Many persons were needed to make the kiln, charge and discharge the kiln, to guard the kiln and to watch the fire, as can be calculated from the achieved working days. In Wijk (The Netherlands) in 1345 during 4 weeks about 700 working days have been counted for men and women to carry; 160 working days were needed to prepare and shovel the lime. (Janse 1981, 165).

Lime trade

Medieval city councils were concerned about the quality of lime. In 1383 an inspection of production and the trade of lime was introduced. In 1531, only lime certified by the city council could be used (Van De Walle 1959, 65).

Rules of the «Bauhutte» in Prague stated that a master mason had to demonstrate to own 7 lime pits.[11] This was to guarantee the quality of the lime.

Even for warfare lime was an important «raw material», especially to construct fortifications. The story is known from Duke Willem II who prepared a battle in Friesland. He shipped out lime to construct compulsion fortifications in Friesland. He didn't succeed and came back with the shipped lime that was sold to the public (Janse 1981, 165).

From a study on the architecture of the Gothic Sint Kwinten church in Leuven (Cuypers 1958) we know that in some cases the church fabric provided the lime to the mason and in some other cases in the same period, the mason of the vaults himself had to provide for the lime [from Namur or Fleurus].

Application of lime mortar.Use of lime mortar

Lime was mixed with sand and as such used as a mortar. Different types of tools were used.

A typical tool used to prepare lime mortar was the lime chopper to mix the sand and the lime and to crush the lime piths (Van De Walle 1959, 82). The lime shovel was used to shovel the lime from the pit to the lime 'sleeve» or basket, with in turn was used to carry the lime to the masons tub. To fill the lime basket at shoulder height it was placed on a tripod or tetra-pod (Du Colombier 1953, Fig. 35).

Different sources indicate that lime was sieved or poured through a great basket with holes (Van De Walle 1959, 82; Salzman 1967, 337) to take out the bigger pieces of burned limestone that probably were badly slaked.

Building and precautionary measures

Masons were divided into different categories. The two more important categories were the master-masons (they were also stone carvers) and the masons. The latter are only responsible for the placing of stones (Du Colombier 1953, 38). Master masons had to teach their apprentices the composition of mortar. The latter were divided in groups as: mortar 'makers', lime 'slakers' and plasterers (Janse 1965, 31). The preparation of mortar was the job of the less skilled workmen. Therefore they were paid as normal workmen (Salzman 1967, 153).

In the winter, masons couldn't work as such due to frost danger of the slow setting lime mortar. In contracts it was often stated which periods of the year allowed a mason to work.[12] To protect masonry against frost in winter it was covered with sod and/or straw and vaults[13] were covered with peat[14] (Janse 1965,.88). In winter the masons were involved in the carving of stone, as it happened in Köln in 1430 (Du Colombier 1953, 37).

Roman mortar in the Renaissance

The Roman mortar technology as described by Vitruvius is taken over in the books of Leone Battista Alberti (De re aedificatoria), Philibert de L'Orme (Ouvrages d'architecture, 1567), Andrea Palladio (Trattato d'Architettura, 1570) and Vincenzo Scamozzi (L'idea dell' Architettura Universale, 1615) (Ferraris 1968).

The influence of the setting on the building process in the Roman period is not well known. Vitruvius (Vitruvius, II° book, chapter IV) only stated that sea-sand slow down the drying process as well as the construction process. Therefore sea-sand can not be used for mortar to erect vaults.

On the other hand Alberti (De re aedificatoria, III° book, chapter 14) is more precise and writes that the building process had to be stopped from time to time to allow the mortar to dry properly.[15] Alberti describes how the centrings of vaults and arches have to be lowered down to guarantee a proportional and smooth setting of the masonry due to the plastic deformation of the mortar. This plastic deformation influences the stress distribution in the arch and in the vaults (Krauss s.d.; Fitchen 1981; Van Balen 1991; Van Balen 2002).

From a report of Raphael to Leo X «on the buildings of antiquity in Rome and the way the ground plans should be measured» of 1519[16] we know that a certain resistance raised against the burning of marble of buildings from antiquity to make lime. This indicates that the recuperation of marble was very usual not only to use it as such (Du Colombier 1953) but also to make lime out of it. The high cost of transport at that time certainly explains this practice.

We prefer to go to the 17th Century to report more progress on the understanding of the use of lime and its hardening.

Lime cycle according to Perrault at the end of the 17TH century.

The comments of Perrault[17] on the writings of Vitruvius' Ten books on architecture give a good idea of the 17th century knowledge on the lime cycle.

Perraults' remarks on the theory of Vitruvius were based on the principles of the iatro-chemical range of thoughts.[18]

The loss of water by which, according to Vitruvius, the limestone loses its strength is, according to Perrault, due to the loss of volatile sulphuric salts (Sels volatiles et sulphurez) during the burning of limestone. Hardening occurs due to absorption (again) of those salts. The increase of strength by mixing lime with sand is due to the exchange of salts between the sand, the stones and the lime. Carbonation can take a long time, as a long time passes before the lime has absorbed all salts that have to return from the stones and the sand.

It is remarkable that he speaks about volatile salts that can be associated with something that has to come out of the air, but the explanation of the hardening refers to the uptake of salts from the stones and the sand.

A HANDBOOK IN PARIS AT THE END OF THE 18TH CENTURY.

In «Le guide de ceux qui veulent bâtir», from the architect N. Le Camus de Mézières (Le Camus De Mézières [1786] 1972) it is shown how, although the chemistry of the hydraulic reactions are known, alchemy inspired the way of working with lime (mortar):

> Une chaux qui est trop longtemps exposée à l'air ou dans un endroit humide s'évapore d'elle-même: le feu et les esprits s'en dissipent, elle se réduit en cendre et n'est d'aucun usage, c'est une chaux 'fusée'.

This handbook gives the best origin of limestone in the environment of Paris (Senlis stone is the best)(Le Camus De Mézières [1786] 1972, 86) and describes the kilns: to burn lime a kiln has to be made with an elliptic inner section (Le Camus De Mézières [1786] 1972, 88). Good limestone can be distinguished from others by the clear sound produced when two slabs are knocked against each other, by the strength and the homogenous milk-white colour. Good quality-limestone yields quicklime that produces twice the volume of lime putty. Lime improves when keeping it for a certain time before use (Le Camus De Mézières [1786] 1972, 87):

> «Il semble que les sels s'aident les uns et les autres; et en effet la chaux est d'autant meilleure qu'elle est plus anciennement éteinte: ne craignez donc pas d'en avoir grande provision du premier instant, exigez-le même de vos entrepreneurs.»

Sand may not be too fine because the mortar becomes to fat by which the contractor will use less lime and the mortar will become to sandy.[19]

Cement is, according to the author, a mixture of lime with crushed ceramics. Good burned clay, e.g. earthenware and tiles, gives the best link with lime (Le Camus De Mézières [1786] 1972, 91).

Mortar has to be made with one third of lime and two thirds of sand everything mixed up very well (« . . . bien broyer, corroyer avec le rabot . . . »). Not too much water may be added, what seems to be difficult (« . . . *vous aurez de la peine à faire valoir ce principe* . . . » (Le Camus De Mézières [1786] 1972, 93). The workability of the mortar depends not only on the amount of water but also on the degree of knocking of the mortar. Lime must be slaked at least a few days before use and must be prepared one day before.

Setting of the mortar can take some years and requires a little bit of humidity, mortar may not dry out as this slows down the hardening « . . . s'il (le mortier) n'étoit pas surpris par la hâle et par une sécheresse trop prompte, il lui faut des années pour se faire, se mûrir, devenir aussi dur que la pierre et s'identifier avec elle . . . » (Le Camus De Mézières [1786] 1972, 94).

The progress of the construction has to take into account the time needed for the setting of the mortar. Still according to «Guide de ceux qui veulent bâtir; . . . » iron bars can be putted beneath the lintels if there is no time to wait for the hardening (Le Camus De Mézières [1786] 1972, 117).

MODERN BINDING MATERIALS FOR MORTAR.

Discovery of the hydraulic reaction.

Many publications have well described the technological evolution caused by the discoveries of Smeaton and Vicat, and placed it in its historical context as (Guillerme 1986; Guillerme 1995)

Smeaton discovered the hydraulic reactions in 1756 searching for a way to make a water resistant lime. From the chemical analysis of the limestone used for the production of natural hydraulic lime he decided:

> the presence of clay in limestone is the most important, if not the only, decisive factor for the hydraulicity.

In 1812 Vicat proved that hydraulic properties were due to the burning together of lime(stone) end clay. Indeed, after de-hydration of the clay and the decomposition of the limestone, a reaction occurs between the quicklime (CaO), the siliceous oxides, the iron oxides and the aluminium oxides. Dependent on the amount of clay and the burning temperature, this reaction is more or less finished and thereof depends the degree of hydraulicity.

The works of Vicat are the basis of all further scientific work on hydraulicity of binders. The result he achieved with his production method is a product that could be placed in between lime hydrate and the actual portland cement. The amount of calcium oxides were so high that this cement had to be slaked. By this slaking calcium oxide has to be converted in calcium hydrate, but too much water had to be avoided, as the hydraulic components may not react with water.

The research methods used by Smeaton and Vicat are evidence of a new scientific understanding. After the philosophy of nature in antiquity (Vitruvius) and the iatro-chemical ideas (Perrault) modern science threw a new insigth on the principals of the hardening of lime and hydraulic limes. The understanding of the complete lime cycle was improved again.

Portland cement.

In 1824 Joseph Aspdin acquired a patent on cement, which he said becomes as hard as portland stone. L. C. Johnson (1835) discovered that the clinker produced by sintering the (hydraulic) lime gave better results when it was powdered. What we actually use as portland cement is the powdered clinker produced by firing the raw material, limestone and clay, to a temperature of 1450°C to which is added gypsum as a reaction retardant. Since the nineteenth century the production of the real Portland cement hasn't changed so much, although a variety of types and admixtures have found their way to the construction market. Scientific research has brought refinements to the production and increased the number of several types of cement for different applications.

In the nineteenth century Belgium was one of the most important producers of hydraulic lime. Limestone was exploited and burned in the region of Tournai. Various old kilns still testify this important industry (Chantry 1979). Today this sources of limestone is used for the production of Portland cement.

From the beginning of the twentieth century Portland cement started to become the most important binder for the fabrication of mortars in masonry and replaced the use of high calcium lime and hydraulic lime in Europe and the United States.

Prescriptions about the use of lime in 19th and 20th century.

A number of prescriptions of nineteenth and early twentieth century give an interesting picture of the use of lime as a binder for masonry mortar.

The Netherlands, in 1843

The Netherlands is known for its history of hydraulic engineering constructions in which they used a mixture of lime and trass. Trass was imported from Andernach in Germany at the border of the Rhine, near Koblenz, but so much associated with The Netherlands so that is was often called «Dutch trass». Dordrecht was an important trade centre of trass and the quality of this trass was controlled and inspected, for a long time, by official inspectors (N 1967).

In «Bouwkundig Magazijn of Schetsen voor Handwerklieden» (N. 1843) different compositions of mortars are given for masonry of different type. They were all based on the use of lime (made of shells or limestone), trass and sand. The amount of trass increased when the masonry mortar was more exposed to water.

The amount of lime (plus trass) to sand was about 1.5 to 2 parts of lime for every part of sand. This ratio is very high and the risk for shrinkage must have been great. We don't know how they managed to keep this under control.[20]

To stimulate carbonation it was advised to make the mortar with more sand in the fall and with more lime in the spring as the mortar had enough time to dry.

When lime was made with shells it was also advised to use more lime in the mortar in case lime made from limestone was used.

Belgium in 1869

From the reading of a handbook of 1869, edited in Mons (Devillez 1869), it becomes clear that the use of cement was much more important in Belgium than in The Netherlands. Here different binders were proposed: high calcium lime, hydraulic lime and cement. The carbonation was described here as a diffusion process that

> slowly progresses from the surface to the centre, as heat progresses in a mass that is heated at the outer side, . . .

Devillez also know that it is important to keep the walls humidified in which hydraulic limes are used. But regularly wetting of the masonry is

> also helpful for mortars with high calcium lime as fast drying can make it pulverised and humidity stimulate the action of carbon dioxide . . .

The sand of lime mortar with high calcium lime may not be to fine to stimulate the diffusion of carbon dioxide. Bastard mortar is described here to be a lime mortar to which cement or pozzolanas are added.

Germany in 1880–1890

In chapter 3 «Die Mörtel und ihre Grundstoffe.» of «Allgemeine Hochbaukunde, des Handbuches des Architektur, 1ste deel, 1ste Band, Die Technik der wichtiger Baustoffe» (Hauenschild 1883) different phases in the increase of strength are described:

> Lime mortar first dries out and strength starts to increase. Afterwards carbonation starts and calcium carbonate crystals are formed.

The porosity of the bricks defines also the amount of water to be added to the mortar: the more porous, the more water has to be added. The author remarks that with the water little lime particles are transported to the contact zone between the mortar and the bricks. This increases the adhesion between both.

Mortars with a low hydraulicity lime and «poor» lime can be slaked with wet sand. This method was much used by the French and the Italians in their hydraulic constructions. Lime cement mortar was the type of masonry mortar used most often. According to Dyckerhoff (Hauenschild 1883) masonry mortars with Portland cement, lime putty and sand have better water resistance, adhesion and are stronger then mortars with equal parts of Portland cement and sand.

The third chapter «Constructions-Elemente in Stein.» from the third part of «Des Handbuches der Architektur» (MARX Erwin. 1886) is also referring to the role of sand: porosity that must allow carbon dioxide to penetrate in the mortar and to induce the carbonation. Sand also provides the surface where calcium carbonate crystals can deposit. Without sand, lime is no adhesive but only a stress distributer.

Lime producers in Belgium at the beginning of the twentieth century.

A technical note in «Bulletin des Métiers et d'Art» of 1909–1910 (N., 1909), gives information about the limestone quarries exploited at that time and the type of lime that can be made with them:[21] «high calcium lime», «weakly hydraulic lime», «moderately hydraulic lime», «hydraulic lime» and «strongly hydraulic lime»

This historical description is ends with the loss of interest in lime after the First World War and the beginning of what we could call modernity in engineering and material sciences.

In Belgium, the fact that the raw materials once used for the production of hydraulic lime [that on its turn was exported to adjacent countries and over the Atlantic] is now exclusively used for the production of cement and the fact that the term hydraulic lime seems to be forgotten, may be seen significant for the trend.

The growing interest in lime today has been boosted by the field of architectural conservation. This is partly due to the many damages noticed in historic buildings due to the use of inappropriate cement mortars in restorations. On itself this is a complex field of research dealt with by different research teams.

Conclusion

The empirically developed technology of using lime expressed in terms of in field application hasn't probably changed so much since the technology was widely established and understood by the Romans. The understanding of the mechanisms responsible for the production, the hardening, and the setting of lime mortar for different applications, has changed together with the evolution of scientific understanding and human's perception of the earth.

An overview of this evolution has been given referring to common lime practice that today is often forgotten. Precious advice on the use of lime for mortar can be thus collected that serves today's research community to look for sustainable building practices and compatible maintenance and repair techniques in conservation.

NOTES

1. Many thanks to Eric Bruehl, research assistant at Getty Conservation for the revision of the English.
2. This paper compiles information from the historical part of the PhD of the author (Van Balen 1991, Ch.2) written in Dutch and never translated into English. Often requests have been received to give distribute its content as to allow scholars to find their way to the content of the work and to the references on the topic. Within the framework of this conference the information was updated. Within the time given in a current sabbatical leave at the Getty Conservation Institute in Los Angeles, other aspect on lime mortar carbonation and its effect on historic structures are being updated and will be published.
3. More recent research in the «New World» seems to indicate that lime can be produced in open fires (Sickels 1996).
4. The hydraulicity index has been defined by Vicat to express to what extent a binder hardens with water. He proved that the presence of calcium silicates and calcium aluminates are determinant for the hydraulicity and defined the index as the ratio of the acids (SiO_2, Al_2O_3 and Fe_2O_3) to the bases (CaO en MgO). The hydraulicity index has no absolute value as it doesn't make the difference between Al_2O_3 and SiO_2, nor between MgO en CaO; it further doesn't give any indication of the bond between the different components which is of great importance to the hydraulicity.
5. This method has also been used by Frizot (Frizot M. 1975; Frizot M. 1981) to characterise Roman mortars. The method can only be used when the sand is siliceous and contains no limestone, as it will be dissolved by the chloric acid.
6. The average of 19 samples gave 30.03% with a standard deviation of 11.42%. From the results of the chemical analyses 8 different groups of mortar could be identified of which 5 groups had more than 1 sample. Grouping of mortars was accomplished taking into consideration different criteria such as: lime content, grain size distribution, hydraulicity index, difference in chemical composition of the oxides and the magnesium content.
7. This method of slaking has been used by the late Prof. R.M. Lemaire for all restoration works after 1952, e.g. at the Great Beguinage in Leuven and the church of Tourinnes-la-Grosse. A mixture of lime and wet sand was kept apart for one year before use. It was then certain that all quicklime was slaked <oral communication from R.M. Lemaire (02/91).
8. Lime kiln: view on the roadstead of Antwerp, 1515; Prentenkabinet Antwerpen; «This is the lime kiln of Flanders» (Van Tyghem 1966, fig.197).
9. According to Burnell —quoted in (Davey 1961)— the amount of fuel to burn 35 cubic feet of lime (about 1 ton) in an intermittent kiln was: 60 cubic feet of oak, or 17 cubic feet of pine, or 9 cubic feet of coal, or 117 cubic feet of peat.
10. An example is known of a claim dating from 1275 regarding the use of 500 oaks from the Wellington forest (Great-Britain) for the limekilns of the king (Davey.1961, 101).
11. Oral communication given by Prof. R.M. Lemaire (†), referring to the still existing copies of the rules of the Prague «Bauhütte». (06/91)
12. At the occasion of the construction of «Walberswick Church» (Suffolk) in 1425: «*Two mason's undertake to build a tower, . . . They shall work yearly from Lady Day to Michaelmas, 'except the first year' (when, presumably, they will be cutting the stone, which could be done at any season)* . . . » (Salzman 1967, p.499).
13. Although vaults were generally built only after the construction of the roof it was considered necessary to cover the vaults to protect them against frost.
14. A clear idea of the protection measures against the frost degradation of masonry can be found in the expenses for the construction of the St. Pieters church in Leiden in 1399. 30 feet reed, 7 ships of peat have been purchased to protect the construction and 1600 sods to put on top of the masonry. This material had to protect newly built walls and pillars against the destructive effect of frost before the construction of the protective roof. It was written down that Jan the «master builder» had to carry peat onto the vaults. In 1625–1627 the expenses of the O.L.Vrouw-over-de-Dijle church in Mechelen mention the costs for a thatcher to cover natural stone and bricks (Janse 1965).
15. An important aspect of the setting and carbonation of lime mortar is quoted here. The theoretical background is still the same as this used by the Romans.
16. Based on the manuscript of Münich, edited by J.D. Passavant(?) in: Raphael von Urbino und sein Vater Giovanni Santi (Leipzig, 1839) quoted in (Choay F. s.d.)

17. (Perrault [1673] 1979), Chapter V. De la chaux & quelle est la meilleure pierre dont elle se fait
18. This is the name given to the era of material (science) that started during the first half of the sixteenth century. Paracelsus was one of the forerunners of this theory. According to this theory all material was composed of three elements: mercury, sulphur and salt. Ontologically speaking, mercury represents the active spirituality, salt represents the passive corporality and sulphur is the link between both (Dijksterhuis 1980, 308).
19. It is good to mention here the complete title of the book of Le Camus de Mézières: 'Le guide de ceux qui veulent bâtir; ouvrage dans lequel on donne les renseignements nécéssaires pour se conduire lors de la construction, *et prévenir les fraudes qui peuvent s'y glisser*» (author's emphasis).
20. Compare with the remarks made by Wisser on the method of mixing high amount of lime with sand (Wisser and Knoefel 1988) as we discussed above.
21. High calcium lime: Rhisne and environment, Arquennes, Ecausinnes, Soignies, Ath (H.), Boussu (H.), Cerfontaine (N.), Ciney (H.), Forries (L.), La Buissière (H.), Maffles (H.), Rochefort (N.), Wépion (N.), Visé (L.), . . . ; Weakly hydraulic lime: Barvaux (L.), and the environment of Durbuy and in Blaton (H.); Moderately hydraulic lime: Altert (L.), Bouvignes (N.), Couvin (N.), Fosses (N.), Horion (L.), Huy (L.), Lavoir (L.), Muno (L.); Hydraulic lime: Antoing (H.), Baelen-lez-Limbourg (L.), Basècle (H.), Calonne (H.), Chokier (L.), Fovrières (L.), Frasnes (N.), Heppignies (H.), Hollogne-aux-Pierres (L.), La Buissière (H.), Wazy (N.), Mevergnies (H.), Oret (N.), Rhisne (N.), Rossignol (L.), Soy (L.), Viesville (H.), . . . ; Strongly hydraulic lime: Chaudfontaine (L.), Chercq (H.), Nismes (H.), Solre-Sambre (N.), Tournai (H.), Vaulx (H.), Antoing («chaux de Coucou»).

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