AN INVESTIGATION ON THE EFFECT OF BRICK DUST ON LIME-BASED MORTARS

John Glengary Carr

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Supervisor
Frank G. Matero

Reader
Jeanne Marie Teutonico

Graduate Group Chair
David G. De Long

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"Mortar a hundred years old is still in its infancy."

Louis J. Vicat in *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural,* (Translated by Captain J. T Smith) 1837.

Introduction

Mortar, an essential material to creating continuous masonry system, has been used in one form or another by many different civilizations, at different times. In antiquity, lime and sand were key ingredients in mortar. However, it seems to have been known by many early builders, masons and architects in the western world that with the addition of a certain quantity of burnt clay, a vast improvement would be obtained in the hardening and hydraulic qualities of the mortar. Hydraulic mortars possess the ability to harden in the presence of water.

There is evidence that burnt clay in the form of crushed potsherds was added to lime mortar to impart it hydraulic qualities in the Minoan civilization of Crete. Similarly, the Romans may have used crushed tile additions to their building mortars before they discovered the material that changed the course of building technology even to the present day. How or exactly when Roman builders discovered a volcanic sand near Naples that when added to lime accelerated setting time and rendered the mortar hydraulic is not exactly known. However, this technology was known to those building at that time and accounts in part for the longevity of their buildings. Vitruvius, the first century architect, builder and writer says of it,

> "There is a species of sand which, naturally, possesses extraordinary qualities. It is found under Baiæ and the territory in the neighbourhood of Mount Vesuvius; if mixed with lime and rubble, it hardens as well under water as in ordinary buildings"[1]

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[1] Vitruvius, *The Ten Books of Architecture*, M. H. Morgan trans., (New York: Dover Publications, 1960) 19.

This extraordinary material referred to by Vitruvius is known as pozzolana, found close to the town of Pozzouli, near Naples, thus the derivation of the name. However, when pozzolana or volcanic earth was not available, Roman builders made use of powdered tiles or pottery or pounded bricks, known as artificial pozzolanas.[2] This material is also referred to as a pozzolana because the resulting mortar has similar properties to that made with natural pozzolana. Such a substitution resulted in hydraulic and rapid setting mortars. Of this Vitruvius said,

> "if to river or sea sand, potsherds ground and passed through a sieve, in the proportion of one-third part, be added, the mortar will be the better for use."[3]

In Roman masonry structures constructed throughout Europe, dust from either bricks or fired clay pots have been found in the lime mortar.

Although at first pozzolana referred only to the material found near Pozzuoli, in time the term came to be applied to other deposits of volcanic ash in Italy, Greece, France and Spain. Still later, pozzolana was used to designate any natural or artificial material possessing properties similar to those of the ash from Pozzouli regardless of its origin.

For the past three hundred years, research has been conducted on the materials responsible for the longevity of Roman buildings. On the subject of pozzolana, the research has determined that whether in the natural or artificial form, when reduced to a powder, and mixed with lime and sand, it

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[2] F. M. Lea, *Investigations on Pozzolans*, (Garsten: Building Research Technical Paper 27), 1940, 4.

[3] Vitruvius, *The Ten Books of Architecture*, M. H. Morgan trnas.,, 9.

displays the property of not only attaining much greater resistance to atmospheric influences, but also the quality of hardening underwater. As well it will impart to the material an increased degree of resistance to various agencies which are normally liable to cause disintegration.

This rediscovery in eigthteenth century Europe permitted the building of maritime structures such as the Eddystone Lighthouse and contributed to the discovery of Portland Cement. Pozzolanas have impacted and continue to impact the way in which building and the repair of buildings is conducted.

Not all brick dust possesses pozzolanic properties. It is not possible to determine the pozzolanicity of a material without testing it in combination of another material such as lime or cement. Microscopic, chemical or mechanical tests can not be performed to evaluate the pozzolanicity of a particular brick dust. The fundamental property of a pozzolana is its ability to chemically combine with lime. Thus a potential pozzolana must be tested in combination with lime to identify and understand its properties.

Although it requires many to years to properly observe the properties imparted on lime mortar with the addition of a pozzolana, this research attempts to evaluate aspects of this phenomenon over a short period of time. In this time period, many factors of the phenomenon can be observed and discussed. Almost a half century ago the following statement was made.

> "The chemistry of pozzolanas is still not solved... and only when the chemical action is completely understood will it be possible to design a pozzolana of ideal composition for any particular purpose."[4]

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[4] R.H. Brogue, *The Chemistry of Portland Cement*, (New York: Rheinhold Publishing Corp. 1947).

Renewed interest in the phenomenon of the pozzolanic reaction is exemplified by the Smeaton Project, a joint venture between English Heritage, ICCROM and Bournemouth University. The investigations of the first phase of the Smeaton Project have examined the use of brick dust as a pozzolanic additive to lime based mortars used in the repair and conservation of historic structures. The first phase of the investigation set out to discover trends in the behaviour of modified lime based mortars and concluded that the addition of brick dust did significantly alter the properties of lime mortars in regards to strength and durability.[5]

This research program intends to apply a methodology similar to that used for the Smeaton Project to investigate the effect of brick dust on the properties of lime based mortars. Like the Smeaton Project, this investigation does not attempt to resolve the mystery of the chemical action of pozzolanas. However, it does attempt to examine and evaluate two potential pozzolanic materials and standardized tests methods. Before the experimental program commenced, a review of the published literature regarding the use of pozzolana was conducted.

The selection of a methodology for evaluating artificial pozzolanas with lime mortar is made difficult by the absence of any generally accepted tests or standards. The difficulty is compounded because pozzolanas themselves have no cementitious properties and thus they must be tested in combination with other materials Thus, pitfalls involved in attempting to

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[5] Jeanne Marie Teutonico, Iain McCraig, Colin Burns and John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars," *APT Bulletin*, Volume XXV, No. 3-4, 32-49.

characterize a pozzolanic material are great as many variables can exist. A standardized methodology was established in an attempt to eliminate many of the variables.

1.1 Pozzolanas and Pozzolanic Reaction

Pozzolanas are currently defined as natural or artificial materials which contain silica and/or alumina that are not cementitious themselves, but when finely ground and mixed with lime, in the presence of water, the mixture will set and harden at ordinary temperatures.[1] There are basically two categories of pozzolanas, namely natural and artificial. Natural pozzolanas are primarily of volcanic origin from geologically recent volcanic activity whereby the material has undergone considerable alteration after deposition.[2] Artificial pozzolanas are either calcined clays or byproducts of various industrial and agricultural processes whereby calcination has occurred.

The fundamental property of a pozzolana is its ability to combine with alkaline lime or cement to yield a material with improved performance. As well, the addition of a pozzolana results in a fundamentally different setting process. Lime mortars modified with either artificial or natural pozzolanas produce a relatively insoluble and durable material that differ from lime mortars. Generally, lime-based mortars require long periods of time to set up or harden and are less resistant to destructive agents including water, freeze/thaw cycles and salts in solution than those modified with pozzolanas.

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[1] ASTM C 593 -89 Standard Specifications for Fly Ash and Other Pozzolans for Use with Lime, 289. Pozzolana, the term used in this research, is also referred to by other sources as pozzolan and pouzzolan. There is no difference, other than spelling, for these terms.

[2] F. M. Lea, Building Research Technical Paper No. 27 *Investigations on Pozzolans, Pozzolnanas and Lime-Pozzolana Mixes,* 1.

Natural Pozzolanas

Both the Greeks and the Romans had discovered that the addition of certain finely ground volcanic deposits mixed with lime and sand would result in a hydraulic mortar with superior strength and endurance. The Greeks still use volcanic tuff called Santorin Earth as a pozzolanic additive to lime mortar. The Romans used and understood that volcanic ash especially from Mount Vesuvius, found close by the town of Pozzouli, would affect the properties of lime based mortars. This region has been worked for centuries with small open pits whereby the material is screened and then ground.[3]

Naturally occurring pozzolanas include some types of volcanic ashes and certain properly calcined opalines, cherts and shales. Another source of natural pozzolana is certain types of diatomaceous earth[4] The occurrence of known suitable natural pozzolanas is limited to only a few regions of the world. The natural pozzolanas known and employed in Europe are the Italian Pozzolanas, German Trass from the Rhine district and Bavaria, Santorin Earth from the Greek island Santorin; Tosca from Teneriffe, one of the Canary Islands, and Tetin from Portugal's Azores. In other parts of the world, natural pozzolanas have been discovered and utilized, such as volcanic ash in Japan. Natural occurring sources of pozzolanas have been sought and located after in North America for use in the concrete industry.

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[3] Alfred Denys Cowper, *Lime and Lime Mortar*, (London, His Majesties Stationary Office, 1927), 47.

[4] Diatomeaous earth has been classified as both a natural pozzolana and an artificial pozzolana. Popovics in *Concrete Materials* lists diatomaceous earth as a natural pozzolana. Wheras, Lea in *The Chemistry of Cement and Concrete* ,16, states that diatomaceous silica is both a natural and an artificial. Some types of diatomaceous earth have no pozzolanic properties at all.

Although many potential sources were located, few if any seem to be utilized for use with lime-based materials.

Naturally occurring pozzolanas are crystalline minerals in particulate form bound firmly together by mutual attraction. The reactivity of clays is related to the type of mineral and the proportions of clay in the material, called the clay fraction.[5] Performance of the clay is improved by heating, thus disrupting the well ordered crystal structure. Temperature and duration of heating is critical as prolonged exposure to too high a temperature can result in re-crystallization and a decrease in reactivity.

Artificial Pozzolanas

Artificial pozzolanas had been known to the Romans, who substituted clays burnt in the form of powdered tiles or pottery for natural pozzolanas. Vitruvius said of this practice

> " if to river or sea sand, potsherds ground and passed through a sieve, in the proportion of one-third part, be added, the mortar will be the better for use." [6]

In the sixteenth century, Biringuccio, made references to the term *opus signinum*, with potsherds, as in the recipe for cisterns.[7] A similar technology, called Surkhi had been known and was used in India. In Egypt, this technology was known as Homra. It has not been established whether the

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[5]W. Mice and J. Allen. *Locating Reactive Natural Pozzolanas* , (Ellis and Moore Consulting Engineers), 3.

[6]Lea, *Investigations on Pozzolans, Pozzolnanas and Lime-Pozzolana Mixes* , 7.

[7] Joan Mishara, "Early Hydraulic Cements," *Early Pyrotechnology, The Evolution of the First Fire-Using Industries*, eds. Theodore and Stephen Wertime, (Washington, Smithsonian Institute Press), 128.

eastern or the western civilization first employed this technology. Surkhi, used for centuries in India, consists of finely ground bricks that replaces the whole or the part of the sand when hydraulic properties are desired. The lime and the brick are mixed wet until a sticky mass is formed, and this is added to the aggregate. The whole mixture is mixed thoroughly and tamped just before usage for masonry construction.

The Romans took their knowledge with them in their Empire, and when no local naturally occurring pozzolanas could be found, they added artificial material in the form of potsherds and pounded bricks. In Roman brick work found in England, artificial pozzolanas thought to be brick dust have been identified in the mortar at Corfe Castle[8]. At the Roman elevated aqueduct in Caesarea, Israel, finely crushed red bricks, tiles or pot sherds were discovered in the multi-layered plaster lining[9] . Ground tiles and potsherds were most commonly used; however, some evidence exists that pozzolanas from Naples were exported for the structures of the Roman Empire.

Analysis of Roman mortars in Germany reveals the increased proportion of fines in the mortar.[10] This increase in fines has been attributed to the addition of brick dust. Brick chippings were used in the mortar as aggregates of varying sizes. The mortar was used for external rendering on the Badenweiler bath ruins; a location where hydraulic mortar and plaster would have been required for its hydraulic properties. The fact that the

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8 *The Builder* , June 18, 1892, 471.

9 Roman Malinowski, "Roman, Concretes and Mortars in Ancient Aquaducts," *Concrete International*, January, 1979,

10 Thorborg Perander and Tuula Råman, *Ancient and Modern Mortars in the Restoration of Historic Buildings*, (Technical Research Centre of Finland, Research Notes, 450), 67.

mortar exists to the present day attests that this method was well under the control of the craftsmen and gave consistent and predictable results.

In 1843 L. J. Vicat experimented with many materials and discovered that properly calcined psammites and schists, smithy slag, and the refuse of the combustion of turf and coal, would yield a hydraulic or pozzolanic set to lime based mortars.[11] At this time Vicat stated that tile dust, "which has been used in buildings for time immemorial, is the most ancient of the artificial pozzolanas known" [12] However, the main artificial pozzolanas are burnt clays and shales, pulverized fly ash, rice husk ash[13], spent oil shales, burnt gaize, burnt moler, and ground granulated blast furnace slag.

Presently the most utilized artificial pozzolanas are burnt clay in the form of brick or tile dust and pulverized fly ash. Pulverized fly ash (PFA), finely ground burnt coal from the furnaces of electricity generating stations which solidifies into spherical particles, is commercially available in Europe and North America for use by the concrete industry. In some cases 35-50 % of ordinary Portland Cement can be replaced by PFA with satisfying results. However, the addition of PFA significantly alters the color, and thus makes its use problematic for conservation.

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[11] L. J. Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural*, translated by Captain J. T. Smith, (London, John Weale 1837), 50. Psammites are geological forms of sandstones. Schists are crystalline metamorphic rocks. Smithy slag are the vitrified byproducts from metal production in the blacksmithing shop.
[12]Ibid., 143.
[13]Calcined rice husks can be classified a pozzolana because a very high of the very high amorphous silica content.

Pozzolanic Reaction

In spite of the considerable research on this subject, the phenomenon of pozzolanic activity is not completely known. As previously mentioned, pozzolanas possess no cementing action without mixing with lime. The addition of this material increases the complexity of this phenomenon. As well, the composition of known pozzolanas and limes can vary widely making it difficult to identify what exactly renders a material pozzolanic. It has been determined that the material must contain silica and alumina which activate the reaction with calcium from the lime in an alkaline or high pH environment.[14] Agreement exists that pozzolanic activity can be described by the following simplified reaction known as C-S-H. Using cement technology notation the main components are C (=CaO), S (=SiO_2), and H (H_2O).

A test developed in 1847 by Vicat can be used as a satisfactory index to pozzolanic reaction. This test, presently adopted as an industry standard worldwide, quantifies the setting time or rate of pozzolanic reactivity to lime mortar. The test produces comparative data to evaluate potential pozzolanas and establish setting rates.

Pozzolanic reactions are not always constant. They are dependent on a myriad of variables including the type of pozzolana, the type of lime, preparation and curing conditions. In the eighteenth century, Vicat established that different types of limes reacted in a clearly different way with pozzolanas to yield different results. Vicat observed that combinations of

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[14]Lea, *Investigations on Pozzolans, Pozzolanas and Lime-Pozzolana Mixes*, 7.

materials could be used to obtain mortars capable of acquiring a great hardness in water, or underground or in situations that are constantly damp[15]. They include:

with rich limes	with slightly hydraulic limes	with the hydraulic limes	with the eminently hydraulic limes
-very energetic pozzolanas, natural or artificial	-simply energetic poz, natural or artificial. -the very energetic pozz. nat. or art. tempered by the mixture of half of sand or other inert substance. -energetic arenes, psammites	-feebly energetic pozz., nat or art. -energetic pozz., nat or art, tempered by a mixture of about one half sand. -energetic arenes psammites	- inert materials such as the quartzose and calcareous sands.* -slag, dross, etc.

* with the eminently hydraulic cements, it was found that the mixture of a highly energetic artificial pozzolana produced a much inferior cement to alike mixture of the same pozzolana with rich slaked lime.

Vicat's observations indicate that many materials, when properly combined, result in what he considered to be a superior hydraulic mortar. An exact explanation does not accompany these observations, and perhaps no acceptable explanation will exist. Presently, many theories for pozzolanic reactions exist[16], although no agreement seems to have been established.

Firing Temperature of Artificial Pozzolana

Although various theories concerning pozzolanic reaction still exist, most academics have agreed that firing temperature has a direct and fundamental effect on rendering the material pozzolanic. It has been

.[15]Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural*, 190.
[16] F. M. Lea, *The Chemistry of Cement and Concrete*, (New York, Chemical Publishing Company, Third Edition, 1971).

established that temperatures ranging from 500° to 950° F will transform certain types of clay into pozzolana. On this subject Lea stated:

> "optimum burning temperature for producing burnt clay pozzolanas can be fixed since the temperature of burning in the rotary kiln of satisfactory materials varied from 775-910 °C. From general experience with both lime and cement mixes it appears that the best pozzolanas are obtained from clays which can be burnt in the upper part of this range."[17]

Other research has produced similar conclusions. In the mid-nineteenth century, Totten published that the best results in terms of resistance came from mortars made with pulverized bricks and tiles which had been lightly calcined rather than those made of more highly burned bricks or tiles. [18] Recently, it has been declared that the best pozzolana is yielded from clay burnt at temperatures between 500 and 900° C.[19] In this temperature range, calcined clays possess pozzolanic reactivity as the crystal lattice of the silicates is destroyed and causes the extreme disorder of the structure. In this state, the amorphous silica becomes more reactive with the calcium hydroxide from the slaked lime, resulting in the formation of an insoluble product which slowly hardens.[20] Thus, heating to high temperatures will destroy the pozzolanic reactivity of some effective materials. Modern brick firing kilns average temperatures considerably higher, roughly 1500°C which renders the material unsuitable as an effective pozzolana. In general, the pozzolanic activity of burnt clay is optimum at

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[17]Ibid., 418.

[18]J. G. Totten, *Essays on Hydraulic and Common Mortars and on Limeburning, Translation of General Treussart, M. Petot and M. Courtois*, (Philadelphia, Franklin Institute, 1838), 149.

[19]Guilia Baronio and Luigia Binda, "Characterization of Mortars and Plasters from Ancient Monuments of Milan (Italy)," *The Masonry Society Journal*, (January - June, 1988, Vol. 7, No. 1), T28.

[20]Ibid.

temperatures whereby the material would be considered underfired by present day standards.

Although firing temperature has been established as a key factor in pozzolanic activity, it can also be influenced by particle size of the clay, mineralogical composition of the clay and the length of calcination time.

Particle Size

Even though there is an understanding that the pozzolanic activity increases with the fineness of the pozzolana, there still exists some question regarding the effect of range in particle size. Fine grinding of the pozzolana whether artificial or natural has been recommended starting with the writings of Vitruvius. In mortars which have endured for centuries, a range of particle sizes has been discovered. It has been established that a range in particle size has ameliorating affects which render lime mortars hydraulic. Research has established that fine grinding of a calcined clay stimulates the pozzolanic reaction and thus the early carbonation of lime mortars.[21] However, the role of larger particle sizes has yet to be fully researched. One theory is that brick dust ground to larger sizes may have a beneficial effect on lime mortars as a porous particulate.[22] A porous particulate is a solid particle with a large internal porosity and suitable pore sizes to act in a fashion similar to that of an air void. Thus, there is the uncertainty as to whether the larger size particles of brick dust behave as an artificial pozzolana or a porous particulate. In lime mortars, these larger size porous members may

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21 Lea, *The Chemistry of Cement and Concrete,* 372.
22 Ibid., 435.

accelerate the initial carbonation by the release of CO_2 to the subsurface. After initial carbonation, the brick dust may act to increase the degree of permeability as well as porosity of the mortar. Resistance to frost and salt damage improves with higher levels of porosity. As lime mortars experience carbonation and the pozzolanic reaction continues for a long period of time, the larger size particles act to absorb and slowly release CO_2 and H_2O to facilitate the reaction. Thus, it could be stated that a range of particle size imparts different but favourable factors for lime mortars.

Mineralogical Composition of the Clay

As previously stated the composition of known artificial and natural pozzolanas can vary widely, making it difficult to identify what exactly renders a material pozzolanic. It has been determined that the material must contain silica and alumina, the principal components of most clays. Generally, most clays are made up of hydrated alumina silicates with about 10-15% water content.[23] Upon firing, the water content is lost and the calcined clay is rendered pozzolanic.

Length of Calcination

Depending on the mineralogical composition and the ultimate firing temperature, the length of calcination can effect the pozzolanic properties of the material. Some materials, such as kaolinite type clays require longer

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[23] Ibid., 420.

periods of calcination.[24] However, prolonged periods of calcination can actually decrease or eliminate the pozzolanic activity of a material.

[24] Ibid., 421.

1.2 Chronology

The following chronology does not pretend to be exhaustive. Instead, it attempts to pull together information from the published research on the lime, cement, concrete and grouting industries. This chronology includes the dates of publications, patents and treatises as well as pertinent projects where the hydraulic properties of mortar or cement were desired. This chronology demonstrates the discovery and rediscovery of pozzolanic materials, the renaissance of pozzolanas and hydraulic lime, followed by their replacement by other patented hydraulic materials such as Portland Cement. Interestingly it was the need to understand and maximize the hydraulic qualities of limestone that led to the invention of Portland Cement. Although the use of brick dust or calcined clay as pozzolanic materials was the impetus for the literature search, other directly related developments are included in the chronology.

Roman Period

The first written material regarding the technology of lime-based mortars with hydraulic additives dates from the Roman period. Mortars were used in combination with hydraulic admixtures such as brick dust, pozzolanic earth and trass in other cultures such as the Greek, Egyptian, and Indian. However, it is from the Romans that hydraulic mortars have been inherited, both through written and physical evidence.

25 B.C. *The Ten Books on Architecture* by Vitruvius is published, containing a section on the use of pozzolanas as a building material.[25]

79 A.D. Mount Vesuvius erupts covering Pompeii and the environ with another layer of volcanic sediment.

138 AD - Hadrian's Wall, constructed with lime and calcined clay particles is completed by the Romans.[26]

532 - Construction of the Hagia Sophia is begun. Analysis of the mortar has concluded that brick dust and chunks of brick were added to the lime based mortar.[27]

Middle Ages

In the Middle Ages following the decline of Rome's power, the art of making hydraulic mortars seemed to be lost as physical or written evidence of the use of this technology has not survived. Some structures may have been constructed using this technology, but it is generally believed that it was not common building practise in Europe.

1000 A.D. - Corfe Castle was constructed. Analysis of mortar revealed that brick dust was included as a hydraulic additive.[28]

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[25] Vitruvius, *The Ten Books on Architecture*, M.H. Morgan trans., (NY:Dover Publications, 1960).

[26] D. L. Rayment and K. Pettifer, "Examination of Durable Mortar from Hadrian's Wall," *Materials Science and Technology*, 1987, 293.

[27] R. A Livingston, R. Marks and M. Erdik, "Analysis of the Masonry of the Hagia Sophia Basilica in Istanbul," *Materials Research Society*, Spring Meeting, San Francisco, CA, May, 1992, and R. A. Livingston and P. E. Stutzman, "Materials Science of the Masonry of the Hagia Sophia Basilica, Istanbul," *Proceedings of the Sixth North American Masonry Conference*, Philadelphia PA, Vol. 1, 1993, 49.

[28] *The Builder*, June 18, 1892, 471.

1290 - one of the earliest found uses of the word mortar - Oxford English Dictionary.[29]

13th C. - In the thirteenth century in England, lime plaster was used on the interior and exterior of buildings after an edict by King John. It has been noted that pounded tiles were added as an aggregate, thus unwittingly imparting a pozzolanic effect.[30]

Rediscovery in the Western World

The use of hydraulic additives to mortar was rediscovered in England and France in the sixteenth and seventeenth centuries and became progressively widespread across Europe. Research on the Roman techniques of lime and concrete construction led to the understanding of hydraulic lime and the eventual discovery of Portland Cement. Until the development of natural cements, the only hydraulic cements were those composed of hydraulic lime or a mixture of pozzolana and lime. However, traditional Indian building methods demonstrate an appreciation for pozzolanic additives in the form of surki or burnt clay to lime mortars, which had been used locally for centuries.

16th C. Vanoccio Biringuccio, an Italian, wrote *Pirotechnica*.. The text discussed the addition of pozzolanic sands to lime mortar to achieve a hydraulic material.[31]

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[29]F. M. Lea, *The Chemistry of Cement and Concrete*, (New York, Chemical Publishing Company, Third Edition), 5.

[30]Alfred Denys Cowper, *Lime and Lime Mortars* (London, His Majesties Stationary Office 1927), 5.

[31] Joan Mishara, "Early Hydraulic Cements," *Early Pyrotechnology, The Evolution of the First Fire-Using Industries*, eds. Theodore A. Wertime and Stephen F. Wertime, (Washington, Smithsonian Institute Press, 1982), 128.

1600 - A recipe for fine lime plaster during the period of the famous Nonesuch Royal Palace near Sutton, was

> "take three parts of pounded Parian marble, add one part of lime which is to be perfectly slaked by letting it lie in a heap covered with pozzolana and exposed to the sun and rain for at least a year. Mix a day before with sufficient water on a tile floor."[32]

mid 18th C. - Baggé of Gothenburg, Sweden experimented with burnt clay pozzolanas for hydraulic projects. He heated schist, powdered it, mixed it with lime and determined that the mortar had the same properties as mortar made with pozzolana.[33]

1756 - John Smeaton (1724 - 1792) was called upon to build a new lighthouse on Eddystone Rock. He made inquiries as to the best materials and discovered that the mortar made from limestone containing a considerable proportion of clay gave the best results. This led him to discover the properties of hydraulic lime. Smeaton experimented with artificial and natural pozzolanas. He used brick dust, powdered forge scales and slag.[34] The lighthouse was built with blue Lias hydraulic lime from Aberthaw mixed with pozzolana from Civita Vecchia in equal quantities.[35]

1774 - *Practical Essay on a Cement and Artificial Stone*, by M. Loriot. Included a hydraulic mortar recipe consisting of one part brickdust finely sifted, two parts of fine river sand and one part old slaked lime.[36]

1776 - *Treastise on Building in Water*, by G. Semple is published in Dublin.

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32 Cowper, *Lime and Lime Mortars*, 5.

33 J. G. Totten, *Essays on Hydraulic and Common Mortars and on Limeburning*, translated from the French text by General Treussart, M. Petot and M. Courtois, (Philadelphia, Franklin Institute), 62.

34 L. J. Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural*, translated by Captain J.T. Smith (London, John Weale, 1837), 50.

35 Lea, *The Chemistry of Cement and Concrete*, 5.

36 Totten, *Essays on Hydraulic and Common Mortars and on Limeburning*, 32

1777 - de la Faye published *Recherches sur la preparation sur la theorie de la chaux dont ils se servient pour leurs constructions, et sur la composition et l'emploi de leurs mortiers* in Paris. In this memoir the author stated that the secret to the durability of Roman mortar laid in the mode of slaking the lime.[37]

1778 - Faujas de Saint-Fond (1741- 1819) published *Recherches sur la pouzzolane, sur la thèorie de la chaux, et sur la cause de la durete du mortier* in Grenoble and Paris. He discovered a naturally occurring pozzolana near the extinct volcanoes of Vivarais, France, and claimed that they equalled those from Naples.[38]

1780 - Bryan Higgins published *Experiments and Observations made with view of improving the art of composing and applying calcareous cements and of preparing quick-lime: theory of these arts; and specifications af the Author's cheap and durable cement for Building, incrustation or Stuccoing, and Artificial Stone* in London. Higgins' research into the particle size of volcanic terra for use with lime indicated that finer particles had more effect at rendering the mortar hydraulic than coarser particles.[39]

1780 - T. Bergman (1735-1784) a Swedish Chemist, after analyzing a limestone yielding hydraulic properties, found that it contained maganese and concluded that this element imparted hydraulic properties to lime.[40]

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[37]Ibid., 96.

[38]Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural*, 48.

[39] Bryan Higgins, *Experiments an dObservations made with the view of improving the art of composing and applying calcareous cements and of preparing quicklime: Theory of these arts, and specifications of the author'scheap and durable cement for building incrusaion or stuccoing and artificial stone*, (London, T. Cadell, 1780), 124.

[40] Jasper A Draffin, "A Brief History of Lime, Cement, Concrete and Reinforces Concrete," *Journal of the Western Society of Engineers* (Chicago, Volume 48, No. 1, March 1943), 6.

1786- Mr Chaptal repeated the experiments of Faujas de Saint -Fond, on the pozzolanas of Vivarais, and claimed that they were inferior to those of Italy.[41]

1788 - Belidor, published *Architecture Hydraulique* in Paris. He recommended the use of pozzolana or trass whenever available for water resistant mortars or plasters. He also recommended the mixture of tiles, stone chips and scales from the a blacksmiths forge, carefully ground and washed of coal, seived and added to freshly slaked lime as a substitute for pozzolana or trass.[42] Belidor gave the name of béton to lime which had the quality of hardening in water.[43]

1791 - *Narrative of the Building of the Eddystone Lighthouse* by John Smeaton is published. This work included the results of the experiments for the selection of materials for the construction of the lighthouse. This work is cited as the first research addressing the elements which increased the strength of lime mortar and permitted it to harden under water.[44]

1791 -Count Chaptal (1756-1852) of France and Switzerland published results of experiments with burnt clay from Languedoc, France. He likened their behaviour and performance to Italian pozzolanas.[45]

1796 - Parker patented a hydraulic cement made by calcining nodules of argilliceous limestone. The patent, number 2170, was taken out in London.[46]

1796 - Lesage, Fench Military Engineer, produced a hydraulic cement from pebbles found at the beach at Boulogne-sur-Mer.[47]

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[41] Totten, *Essays on Hydraulic and Common Mortars and on Limeburning*, 62.
[42] Lea, *The Chemistry of Cement and Concrete*, 5.
[43]Totten, *Essays on Hydraulic and Common Mortars and on Limeburning*, 3.
[44] Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 6.
[45] Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural*, 182.
[46] Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 7.
[47] Ibid., 6.

1800 - Parker's patented product was given the name Roman Cement.[48]

1802 - Charles Berigny successfully grouted a sluice at Dieppe, France with a mixture of Italian pozzolanas and lime.[49]

1805 - Rondelet published *L'Art de Batir* in Paris. Rondelet carefully examined Roman mortars for content and theorized on the method of their preparation. He attributed their durability to their long slaking time.

1810 - Dutch Society of Science discussed why lime made from limestone was better than that made from shells and launched the experimention into methods to improve shell lime to produce a better quality lime mortar.[50]

1811 - James Frost first patented a cement product and established works at Swanscombe, England.[51]

1813 - Collet-Descotels (1773-1815) Professor of Chemistry at the School of Mines in France, stated that it is essential for limestone to contain a high quantity of fine grained siliceous material to yield good hydraulic lime mortar.[52]

1818 - J. Louis Vicat (1786- 1861) a French Engineer, published *Reserches Experimentales* in Paris. Vicat investigated the suitablity of of the various French limestones for the production of lime, and stated that lime or cement with hydraluic properties must contain lime, silica or alumina. At this time Vicat invented the method of testing the hydraulic properties of a mortar by time required to set, called the Vicat needle.[53]

48Ibid., 7.

49 *History of Grouting*, 271.

50Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 7.

51 Lea, *The Chemistry of Cement and Concrete*, 6.

52 Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 6.

53Ibid., 7.

1818 - The first natural cement was made in America near Chittenango, Madison County, New York, by Canvass White, an Engineer working on the construction of the Erie Canal.[54]

1818 - The navy dry docks at Rochefort, France were successfully grouted using a pozzolana and lime mortar grout.[55]

1819 - J.F. John (1782- 1847) Professor of Chemistry, published a disseration titled *Lime and Mortar* in Berlin. He concluded that the presence of clay, silica and iron oxide improved the quality of lime for mortars. He independently came to the same conclusion as Vicat.[56]

1822 - James Frost patented "Britsh Cement," an artificial cement whereby the raw material was calcined until all the carbonic acid was expelled, and the material was finely ground.[57]

1824- James Aspdin (1779-1855) took out the first patent for a new and improved natural cement called Portland Cement. It was so named, because, when hardened, it resembled the limestone of the Isle of Portland. Aspin acheived this material by burning argillaceous limestone nodules found in London clay and in the shale beds of the Lias formation[58]

1825 - 1836 - Col. J. G. Totten, Colonel in the United States Army, experimented with lime based, natural cement and patented cement mortars at Fort Adams, Newport Harbour, Rhode Island. He observed that brick dust or the dust of burnt clay, improved the quality of mortars both as to durability and hardness. Hydraulic cement, burnt clay, or brick dust was added to every kind of mortar made at Fort Adams in proportions varying with the purpose

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54 Ibid., 8.

55 *History of Grouting*, 271

56 Lea, *The Chemistry of Cement and Concrete*, 6.

57 Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 8.

58 Lea, *The Chemistry of Cement and Concrete*, 6.

to which the mortar was to be applied. Totten experimented with concrete mix consisting of lime, sand and brick fragments and granite fragments. He did not publish the results of these experiments until 1838.[59]

1826 - Sir Charles Pasley (1780-1861) started to research the effect of firing temperature and vitrification of the calcined clay added to produce hydraulic mortar and cement.[60]

1828 - Portland Cement is experimented with for the construction of the Thames River Tunnel in London, but is only used in a limited capacity.[61]

1828 - Vicat published a treatise on his experiments.

1828 - Cement works were established at Rosendale, Ulster County, N.Y. Rosendale Cement was quarried from magnesium limestone deposits, and was one of the most widely used and long lived commercial natural cements in North America.[62] Experimentation and development of natural cement at Rosendale was directly related to the construction of the Erie Canal.

1829- Limestone deposits producing good quality natural cement were discovered and utilized near Louisville, Kentucky [63]

1837- Vicat's *A Praticial and Scientific Treatise on Clacareous Mortars and Cements, Artificial and Natural* was translated into English by Captain J. T. Smith.[64]

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59 Totten, *Essays on Hydraulic and Common Mortars and on Limeburning*. 238 and 231.

60 Sir Charles Pasley, *Observations on limes, calcareous cements, mortars, stuccos and concrete; and on pozzolanad natural and artificial*, (London, J. Weale, 1838), 187.

61 Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 11.

62 Ibid., 8.

63 Ibid.

64 Vicat, *A Practical and Scientific Treatise on Calcareous Mortars and Cements, Artificial and Natural.*.

1837 - Raynal wrote a paper on the use of grouting for repairing masonry. He recommended the use of hydraulic lime, whereby 2 parts lime were mixed with 3 parts pozzolana and sufficient water to make it semiliquid.[65]

1838 - C.W. Pasley published *Observations on Limes, Calcareous Cements, Mortars, Stuccos and Concrete.*

1840 - First commercial Portland Cement plant in France was established at Boulogne Sur Mer.[66]

1850 - Natural cement works were established at Seigfried Pennsylvania, establishing the Lehigh Valley district as an important cement producing center in North America.[67]

1851 - Isaac Charles Johnson set up cement works at Rochester.[68]

1855 - First commercial portland cement plant is established in Germany at Züllchow near Stettin.[69]

1859-1867 - First extensive use of Portland Cement in a construction project during the construction of the sewage system of London.[70]

1868 - *The Practical Manufacture of Portland Cement,* by A Lipowitz was published, spreading this technology to Germany.

1870 - General Scott took out a patent for selenitic lime and a company was formed to carry on its manufacture[71].

1870 - General Quincy Adams Gilmore (1825-1888) published *Practical Treatise on Limes Hydraulic Cements and Mortars* in the United States.

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[65]*History of Grouting*, 271.
[66] Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete* , 11.
[67] Uriah Cummings, *American Cements*, (Boston, Rogers and Manson, 1898), 19.
[68]Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 11.
[69] Ibid.
[70] Ibid.
[71]Selenitic lime is a lime, usually hydraulic, to which a small proportion of calcined gypsum has been added. This was considered to result in increased strength. From Cowper, *Lime and Lime Mortars*, 78.

1871 - *Practical Treastise on Coignet Beton and Other Artificial Stones* by General Q. A. Gillmore was published.

1871 - Portland Cement became commercially available in the United States from a plant at Coplay, Pennsylvania, operated by David A. Saylor, Adam Woolever, and Esias Rehrig.[72]

1871- Thomas Millen (1832-1907) began to manufacture Portland Cement in South Bend, Indiana.[73]

1874 - Robert W. Lesley (1853-1935) organized a cement selling business in Philadelphia and sold 10, 000 barrels of Portland Cement on his second day of cement sales .[74]

1875 - John K. Shinn, began to manufacture Portland Cement in Wampum Pennsylvania.[75]

1883 - J. N. Fuchs identified that quartz and other forms of crystalline silica are inactive while the amorphous and hydrated silica behave as pozzolanas.

1886 - Jose F de Navarro (1823- 1909) revolutionized the cement industry by introducing an inclined rotary kiln capable of producing 160 - 300 barels a day[76]

1887 - *Experimental Researches on the Constitution of Hydraulic Mortars*, by H. LeChatelier, was published, but by this time, the use of Portland Cement has begun to dominate construction practices

1888 - *Notes on the Compressive Resistance of Freestone, Brick Piers, Hydraulic Cements, Mortars and Concretes* by General Q. A. Gillmore was published in New York.

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[72]Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete*, 11..
[73] Ibid.
[74]Ibid.
[75]Ibid.
[76]Ibid., 12.

1893 - *Manual on Lime and Cement* by A.H. Heath is published in New York.

1898 - *American Cements,* by Uriah Cummings was published in Boston.

1902 - Burnt clay was effectively used as a pozzolana in the construction of the Asyût Nile Barrage.[77]

1909 - Mr. White published in the *Journal of English Industrial Chemistry,* that a micro-chemical test based on phenol as a reagent could effectivly detect quicklime in the presence of slaked lime.[78]

1909 - Thomas A. Edison, produced a rotary kiln capable of producing 1000 barrels a day.[79]

1909 - C.J. Potter described the process of mixing ground burnt clay with Portland Cement yielding Potter's Red Cement, for use in freshwater and seawater construction.[80]

1914 - Documented report of fly ash being used as a pozzolanic material in concrete.[81]

1919-1925 - Construction of the Sennar Dam on the Blue Nile, used cement composed of 70% Portland and 30% burnt clay produced on site.

1937 - R. E. Davis et al. U.S. studied the use of Pulverised fly ash for use in concrete.

1939-1945 - Revived use of burnt clay to Portland Cement during World War II as an economic measure.[82]

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[77]Cowper, *Lime and Lime Mortars,* 48.

[78]*The Builder,* (April 23, 1922), 663.

[79] Draffin, *A Brief History of Lime, Cement, Concrete and Reinforced Concrete,* 12

[80] Lea, *The Chemistry of Cement and Concrete,* 420.

[81]Richard Helmut, *Fly Ash in Cement and Concrete,* (Skokie Illinois: Portland Cement Association, 1987), 2.

[82]*Symposium on the Use of Pozzolanic Materials in Mortars and Concrete,* San Francisco, 1949. (ASTM, Philadelphia, 1950)

1940 - F. M. Lea, working in the United Kingdom, commences research on the production, properties and the ulitilisation of pozzolanas manufactured by the burning of suitable clays and shales.

1940's - Established that the addition of Pozzolana to concrete will eliminate or greatly reduce the effects of alkali-aggregate reaction.[83]

1949 - ASTM Symposium on Pozzolanic Materials held in San Francisco.

1981 - ICCROM International Symposium on Mortars, Cements and Grouts used in the Conservation of Historic Buildings is held in Rome. First large scale attempt at the organization of a scientific approach to the problem of mortars for repair.

1987 - Smeaton Project in England establishes a testing program to contribute to the understanding of the characteristics and behaviour of lime based mortars for the repair and conservation of historic buildings.

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83 R. Mielenz, L. Witte, and O. Glantz, *STP 99*, (ASTM, Philadelphia) 45.

1.3 Review and Assessment of Published Literature on Lime-Pozzolana Mortars for the Repair of Historic Structures

Interest in mortars from the point of view of repair and conservation of historic structures is relatively recent. The first attempts to characterize and standardize repair mortars dates to 1981, on the occasion of the International Symposium on Mortars, Cement and Grouts used in the Conservation of Historic Buildings held at Rome.

At this time three fundamental parameters were identified. They include[84]:

1) Research should be carried out in parallel on both new and ancient mortars. Restoration mortars must be prepared taking into account the characteristics of the materials to which they are applied or which they substitute.

2) New mortars for restoration should be characterized clearly, by identifying certain fundamental parameters.

3) Methods for measuring these parameters should be standardized.

Since that seminal symposium, the formulation of repair mortars by those in the field of architectural conservation usually involves identification of the properties and constituent parts of the original mortars coupled with

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[84]P. Rota Rossi-Doria, "Mortars for Restoration: basic requirements and quality control," *Matériaux et Constructions,* Vol.19, No. 114, 1986, 445.

the examination of the physical and mechanical properties of the repair mortar. This first step of examination of the constituent parts of the original mortar has resulted in the identification of natural and artificial pozzolanas. For the formulation of a repair mortar, researchers have investigated the properties of lime based mortars modified with both natural and artificial pozzolanas. The following review and assessment of selected published research will identify the objectives, the materials, the examination methods, and the results of each investigation.

The Smeaton Project

The Smeaton Project,[85] a joint research program of ICCROM, English Heritage and Bournemouth University, grew out of experimental work to identify suitable mortars for use in the conservation of Hadrian's Wall. Samples of jointing and core mortar samples were found to contain lime, crushed tile, crushed sandstone, sand and kiln debris, as well as some animal fat, probably tallow.[86] In the first phase of this research project, the broad objectives were to "contribute to the understanding of the characteristics and behaviour of lime-based mortar by attempting to identify - and where possible quantify - the material and practice parameters that affect mortar properties."[87] In doing so the experimental program focused on the effects of set additives, specifically brick dust and cements on the performance of lime and sand mortars.

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[85]Jeanne Marie Teutonico, Iain McCraig, Colin Burns and John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars," *APT Bulletin*, Volume XXV, No. 3-4, 32-49.
[86]Ibid., 34.
[87]Ibid., 33.

Brick dust was investigated in order to understand the effects of such factors as optimum particle size, firing temperature, and proportion of brick dust in the mix. The experimental program included moisture content, stiffening or setting rate, compressive strength, depth of carbonation, sodium sulphate crystallization test, as well as monitoring exposed samples at regular intervals.

The results of the first phase of the project concluded 1) that the addition of brick dust does significantly alter the properties that were tested, of lime mortars, 2) low-fired brick dusts seem to have the most positive effect on the strength and durability of the mixtures, 3) the addition of small amounts of cement to the mixtures has a negative effect on the strength and durability of the mortars. Similarly, it was found that a particle size ranging from <75 microns results in the brick dust acting as a pozzolana and that a firing temperature below 950°C produces the best quality brick dust for the addition to lime mortar.[88]

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[88]Ibid., 42.

Research at Aristotle University of Thessaloniki, Greece

In three published works from the Aristotle University of Thessaloniki[89], [90], researchers have examined the role of both artificial and natural pozzolanas in mortars and grouts for the repair of historic masonry structures. The aim of these research projects was to determine the composition and proportions of repair mortar and grout which will compatible to the materials used in the historic structures in Thessaloniki from the 4th to the 15th century A. D.

In a paper entitled "Pozzolanic Mortars for Repair of Masonry Structures," mortars from historic structures were examined and determined to contain sand, lime and in some, fragments of powdered brick. Those that contained hydraulic components such as pozzolana or brick dust, determined by X-ray analysis, were stated to have a higher compressive strength than those without hydraulic components. To demonstrate the contribution of pozzolanic additives to strength development of the mortars, physical tests were conducted. These tests included compressive strength, tensile strength and modulus of elasticity. Although this research focuses on the mechanical properties of the lime based mortars, others properties of lime based mortars have been included in this methodology. The results of the research indicate

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89 Penelis, G., Papayianni, J, and M. Karaveziroglou. "Pozzolanic Mortars for Repair of Masonry Structures," *Structural Repair and Maintenance of Historic Buildings*, (Boston: Computational Mechanics Publications, 1989), 161-169.

89 Penelis, G., Karaveziroglou, M. and Papayianni, J. "Grouts for Repairing and Strengthening Old masonry Structures" *Structural Repair and Maintenance of Historical Buildings*, (Boston: Computational Mechanics Publications, 1989), 179-188.

90 Karaveziroglou-Weber, Maria and Papayianni, Ioanna. "Long-term Strength of Mortars and Grouts Used in Interventions" *Structural Preservation of the Architectural Heritage, Report of the IABSE Symposium*, (Rome, 1993), 527-532.

that appropriate gradation and a particle size maximum of 2 mm for the pozzolanic additives increases the mechanical strength of the mortars.

The fundamental problem associated with this research is the predisposition to include cement as an additive to the lime based mortars in order to achieve increased mechanical strength. The results of this research indicate that the compressive strength of lime-sand-pozzolana mortars was equal to some of the mortars with lime- sand-cement in small proportions. This research seems to overlook the problems associated with the addition of cement, even in small proportions to lime-based mortars.

In a paper titled "Long-term Strength Development of Mortars and Grouts used in Interventions," the same principal researchers have set up a testing program for "traditional materials" including lime, natural pozzolana, brick powder and crushed bricks. In this paper, the researchers have stated that pozzolanic reaction follows a slow, time-dependent process. In addition to these traditional materials, high proportions of cement has been added to the mortars and grouts being evaluated. Not surprisingly, those mortars modified with cement mortar exhibit higher values of compressive strength, tensile strength and modulus of elasticity over a period of four years. Again, the evaluation of strength should not be the only criteria for which to judge a lime-based mortars.

Research at University of Seigen, Germany

In a short paper delivered at the 1988 International Stone Conference[91], researchers discussed the use of microscopical methods to understand the interactions of mortar components. In this research, historic lime-based mortars known to contain brick dust and pozzolana were compared to lime -based mortars made with commercially available hydraulic products such as diatomaceous earth, condensed silica fume, fly ash and blast furnace slag. Using microscopical methods, lime mortars modified with various admixtures were examined with the electron microscope to compare the reactions. The researchers identified and determined that C-S-H fiber did exist in historic and replicated mortars. However, in the historic mortars, redepositions of calcium carbonate were visible in some of the microcracks, effectively waterproofing the surface. It was noticed that some artificial pozzolanas from industrial processes had the tendency to effloresce.

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91 Wisser, S.; K. Kraus, and D. Knöfel. "Composition and Properties of Historic Lime Mortars," *Proceedings of the VIth International Congress on Deterioration and Conservation of Stone,* (Torun, Poland, 1988), 484-491

1.4 Desirable Properties of a Mortar for the Repair and Conservation of Historic Structures

Generally, mortar is the most frequently repaired component element of a masonry system. In most cases, the repair of deteriorated mortar involves replacement. The approaches to mortar replacement include: 1) replacement in kind; 2) replacement with modern materials that are similar to the properties of the original mortar; and 3) replacement with modern materials that are deemed "better" than the originals. For replacement in kind or with similar modern materials, mortar characterization must be conducted. Even if characterization of the original mortar has been conducted, few preliminary tests are conducted to evaluate the properties of the repair material.[92]

For the selection of a repair mortar, the parameters and behavior of the new mortar must first be understood. First, the purpose or role of the mortar must be defined, as differences in properties and constituencies can exist between bedding and pointing mortars. Similarly, the desired properties of a repair mortar must be understood. Generally the following are desired properties of a mortar for the repair and conservation of historic structures.

1) Good workability, as defined by both the mortars' ability to be manipulated by masonry tools and its cohesiveness and adhesion to the masonry unit to form a well packed continuous mass.

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[92]P. Rota Rossi-Doria, "Mortars for Restoration: basic requirements and quality control," *Matérial et Constructions*, Vol. 19, No. 114, 446.

2) A consistent and reliable setting rate whereby the initial set of the mortar will not cause delays to the repair or conservation work.

3) Low or no shrinkage of the mortar to reduce microcracking or cracking at the interface of the masonry unit.

4) Elasticity. As masonry systems are often subject to movement, the mortar should act to cushion the masonry unit without cracking or causing cracking to the masonry unit.

5) Relative strength as related to the strength of the masonry unit and the masonry system.

6) Water and water vapor permeability to reduce water or water vapor from being trapped and freezing in the masonry system.

7) Resistance and durability to the increase of liquid water. This property is related to the open and closed pore sizes of the outer zone of the mortar.

8) Resistance to salt attack or other deleterious solutions. This property is related to the pore sizes and distribution in the masonry system.

9) Retreatability in that the mortar as a repair material should be a sacrificial component of the masonry system which can be easily removed without causing damage to the masonry unit.

2.1 Research Significance

A review of the literature dealing with the addition of artificial pozzolanas to lime-based mortars reveals a rich source of information on usage yet few explanations on performance. Included in the technological literature are the historical developments, ingredients and uses of these mortars. In recent conservation literature reviewing historic mortars, careful consideration is given to the ingredients, uses and appropriateness for a repair material. However, what is generally lacking is a quantitative description of the composition of mortar and the affect that composition has on overall physical, mechanical and chemical characteristics. The Smeaton Project has been set up in to contribute to the understanding of the characterisitcs and behavior of lime-based mortars by attempting to identify and where possible quantify the materials and practice parameters that affect their properties.[93]

This experimental program attempts to act as a compendium to the Smeaton Project by examining the characteristics and behaviour of the components of traditional mortars made with North American materials. By examining the mortar characteristics that concerned the Smeaton Project, it is intended that the research presented in this experimental program will produce some comparable data on the materials and practice parameters of lime-based mortars modified with brick dust.

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93 Teutonico, "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars, " 33.

In addition to shedding light on lime mortars modified with brick dust in the North American context, the methodology presented in this study has the potential for further implications and applications. These include:

1) Application of the experimental testing program to other potential artificial pozzolanas for use with lime or cement. These include bricks salvaged from construction sites, pulverized fly ash, calcined clay, rice husk ash and ground granulated blast furnace slag.

2) Reconsideration of the standards available for the evaluation of mortars used in the repair of historic masonry structures so that mortars replicating traditional ones will not fail to meet these standards.

3) Application of the testing program on lime and pozzolana traditions as an appropriate technology for developing nations whereby lime with hydraulic additives could be an affordable and renewable material. This would reduce the need for Portland Cement, which is more expensive and often depends on importation. In countries where Portland Cement is expensive or in short supply, pozzolanas, such as brick dust have been and could be substituted to up to 40% of the total mixture without significantly reducing the quality of the final product.[94]

4) To help reduce environmental pollution. Portland Cement production requires a lot of pollution producing energy, so reducing its

[94]*Appropriate Building Technology,* 65.

use when appropriate would be environmentally sound. As well, many potential pozzolanic materials are by products of the manufacturing, agricultural or construction industries. By using these byproducts, such as fly ash, salvage historic bricks, or corn husks, land fill space can be reduced.

2.2 Research Objective

In broad terms, the goals of this study are to contribute to an understanding of lime-based mortars for the repair of historic structures in North America. In more specific terms this examination will attempt to address research questions ranging from evaluating the materials to testing standards and methods. The objectives of this experimental program include:

1) To observe, evaluate and when possible, quantify the affect of the addition of two types of brick dust to the properties of lime-based mortar through an experimental program of physical and mechanical testing.

2) To understand and appreciate the phenomenon of pozzolanic reaction as evidenced by the addition of two types of brick dust to lime-based mortar.

3) To evaluate and compare the phenomenon of pozzolanic reaction to that brought about by a porous particulate as evidenced by the addition of brick dust and limestone dust to lime-based mortar through an experimental program of physical and mechanical testing.

4) To observe, evaluate and quantify the behaviour of materials presently being used in North American repair practices.

5) To establish a testing program for the evaluation of prospective materials for use in the repair of historic structures.

6) To review, evaluate and comment on the appropriateness of North American testing standards for lime-based materials.

7) To identify research priorities and possibilities regarding the effect of the addition of brick dust to lime-based mortars.

2.3 Materials

The selection of materials used in this study was based on two main criteria. The first was that the materials be commercially available in North America. The second was the availability of information regarding the chemical and physical properties of the material. Although one of the two brick dusts examined is not commercially available, it was selected because information regarding some of the physical and mechanical properties was available. The materials used reflect standard contemporary conservation practice.[95] The lime putty and sand were constant throughout the study. To these materials were added two different types of brick dust. For one aspect of the study, limestone dust was added to the lime and sand mixture as an inert porous particulate.

Sand

A sharp well-graded commercial masonry sand, Yellow Bar Sand, supplied by Dunrite Sand and Gravel, P.O. Box 681, Vineland, New Jersey 08360, was selected for its compliance to ASTM C778-89 Standard Specification for Standard Sandand because its chemical composition was known.[96] Analysis reveals 99.5% silica dioxide with trace amounts of various other minerals.

95John and Nicola Ashurst, *Practical Building Conservation, Volume 3, Mortars Plasters and Renders*, (New York , Halstead Press), 66.
96 Chemical Analysis for the Yellow Bar Sand was conducted in 1990 by Testwell Craig, Testing Laboratories, Mays Landing , New Jersey.

Table 1 - Particle Size Distribution of Yellow Bar Sand

Sieve #	% Retained	% Passing
4	0	100
8	0.2	99.5
16	4	95.5
30	21.5	74
50	34.7	39.3
100	27.7	11.6
200	9.4	2.2

Lime

Lime used in this study was supplied by Beachvilime Limited, (P.O Box 190, Ingersoll, Ontario, N5C 3K3, Canada) and produced at the Beachville East Plant, Beachville, Ontario. It is obtained by the calcination of high calcium limestone to produce calcium oxide (CaO), commercially available as masons quicklime. Masons quicklime is a calcined limestone capable of slaking with water, and suitable for use for masonry projects. It is considered a High Calcium Lime indicating less than 5% magnesium carbonate was found in the mixture. Generally, commercial hydrated lime available in North America is magnesian lime, with 5 to 35% magsesium carbonate present in the limestone used.

Composition and Physical Properties of Beachvilime:[97]

Chemical -

Calcium Oxide (CaO)	95.5%
Magnesium Oxide (MgO)	1.0%
Silica (SiO_2) and Insolubles	0.75%
Ferric Oxide (Fe_2O_3)	0.15%
Alumina (Al_2O_3)	0.15%

[97]Physical properties supplied by product manufacturer, Beachvilime Limited. Based on ASTM C-110-87 *Standard Test Method for Physical Testing of Quicklime, Hydrated Lime and Limestone.*

Total Sulphur (S)	0.03%
Loss on Ignition	2.5%
Available Lime as calcium Oxide (CaO)	92%
Carbon Dioxide	2%

Physical -

Bulk Density	
Angle of Repose	45 degrees
Specific Gravity	3.4 (relative density)
Solubility in Water	1.3 g/litre @ 20° C
Basicity Factor	0.93
Slaking Rate[98]	
(1) Temp. Rise 30 sec	25 degrees
(2) Total Temp. Rise	48 degrees
(3) Total Active Slaking Time	6 minutes

The slaking of the lime was conducted on May 15, 1993. Slaking of the lime was achieved in a large metal mortar tray (1m x 1m). Water was added to the tray followed by the quicklime. The mixture was slowly stirred with a hoe until the reaction of the lime with the water ceased. A thick white substance called lime putty was the result of this initial slaking process. The putty was pressed through a 2.5 mm or 1 in sieve to remove any lumps of unreacted quicklime and was stored in plastic pails and sealed with lids to prevent carbonation. Approximately 3 cm of water laid on top of the lime putty. The putty was then stored roughly 5 months until the commencement of the experimental program.

Brick Dust

Two types of brick dust were selected and tested for the experimental program. The first type (**Brick Dust 1**) was supplied by Martin Clay Products,

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[98]Based on ASTM C 110-87 *Standard Test Method for Physical Testing of Quicklime, Hydrated Lime and Limestone,* (modified 1:4 lime:water)

Parkhill, Ontario. This material is produced for the construction of clay tennis courts. Underfired bricks considered seconds by brick manufacturers are collected by Martin Clay Products and ground into a powder. Although not manufactured for masonry purposes, this material has been used in Canada for repair mortars where hydraulic properties are desired. This material was selected for the experimental program due to its apparent success as a pozzolana and its commercial availability in North America. The exact firing temperature of these bricks is not known. The exact mineral composition of the brick dust is unknown, although they do contain silica and alumina. Particle size was determined.

The second brick dust (**Brick Dust 2**) used in the experimental program was supplied by Colonial Williamsburg. This material was selected after numerous inquiries to brick manufacturers to locate a low fired brick. Colonial Williamsburg has a brick yard with a kiln used for brick production for repair work done on site. Although this material is not commercially available as an additive, it was selected because of the ideal firing temperature of the clay of 1650° F. or 898.8° C.[99] Research has established that an optimum burning temperature for producing burnt clay pozzolanas varies from 775-910 °C.[100] [101]

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[99] Records of Brick Kiln, 1992, Supplied by Colonial Williamsburg. The firing temperature was monitored by eighteen gauges at different locations in the kiln. The four day firing period achieved the maximum temperature in some locations of 1650° F or 898.8° C

[100] Jeanne Marie Teutonico et al., "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars", *APT Bulletin*, Vol. XXV, No. 3-4, 41.

[101] Luigia Binda and Guilia Baronia state in "Characterization of Mortars and Plasters from Ancient Monuments of Milan (Italy)," *The Masonry Scoiety Journal*, (January-June 1988), T23, that the best pozzolanic activity is yielded from clay burnt at temperatures between 500 and 900°C.

Both BD 1 and BD 2 were ground in a metal grinder and sieved with standard sieves yielding a well-graded product type ranging from 75 to 300 µm. The same amount, measured by volume was taken off each sieve (ASTM #50 to #200) to form the additive. Thus an even distribution based on particle size was established for both types of brick dust.

Stone Dust

Stone dust, derived from crushing and grinding limestone was used in the experimental program. The limestone was supplied from Northumberland County, Pennsylvania, and commonly known as Helderberg Limestone. It is a high-calcium limestone with trace amounts of magnesium, silica and alumina. The composition of this stone was reported as:[102]

96 % $Ca\ CO$
1.5 % $Mg\ CO_3$
1% $Al_2\ O_3 + Fe_2\ O_2$
1% $Si\ O_2$

The ground limestone was sieved and an even amount measured by volume was taken from each sieve to yield a particle size equal to that of the brick dust. A simple porosity test determined the limestone to have a porosity of about 20%.

This limestone dust was selected as an inert porous particulate, as it has 20 % porosity while not reacting chemically with the lime-based mortar.

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102 Benjamin Miller, *Limestones of Pennsylvania*, (Pennsylvania Geological Survey, Fourth Series, 1934), 551.

2. 4 Formulations of Facsimiles

Consistency of the preparation of the facsimile samples for the experimental program was guided by testing standards and a single operator/mixer. This resulted in a high degree of uniformity during the preparation of the samples.

Proportions of the mixes were determined after a review of the historical literature[103], contemporary conservation practice and contemporary experimental work presently being conducted in Europe[104] A comparison of these practices reveals no significant change in proportion over a two hundred year period[105 106].

Proportioning of the mixes was completed by volume. The sand and brick dust were measured dry.

Table 2 - Mortar Facsimile Compositions

Mix	Lime	Sand	Brick Dust 1 (Williamsburg)	Brick Dust 2 (Martin Clay)	Limestone Dust
1	1 part	3 parts			
2	1 part	3 parts	1 part		

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[103]In 1756 during the construction of the Eddystone Lighthouse, Smeaton specified "two bushels of lime powder, one bushel of pozzolana, and one bushel of common sand." resulting in what is 1: 1: 2 mix. Sir Charles William Pasley, *Observations on limes, calcareous cements, mortars, stuccos and concrete; and on pozzolanas, natural and artificial*, (London, J. Weale 1838), 182.

[104] In the Smeaton Project, the ratio of sand:lime: brick dust ranged from 2 1/2:1:1 to 5:1:1. Jeanne Marie Teutonico,et al., "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars," *APT Bulletin*, Vol. XXV, No. 3-4, 36.

[105]Monsieur Loriot 1774, Paris, 32 Called for: Take one part of brickdust finely sifted, two parts of fine river sand screened, and as much old slaked lime as may be sufficient to form mortar.

[106]Penelis, Papayianni and Karaveziroglou, "Pozzolanic Mortars for the Repair of Masonry Structures," *Structural Repair and Maintenance of Historic Buildings*, 165. Twentieth century specification for repair mortar consisting of 1 part lime, 4 parts sand and 2 parts of brick dust

3	1 part	2.5 parts	1 part		
4	1 part	2.5 parts			1 part
5	1 part	2.5 parts		1 part	

2.4.1 Mixing

Test Standard Consulted - ASTM C 305 Test Method for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.

Initial mixing of the roughage, lime putty and sand, was conducted in a laboratory but in a manner similar to masonry practice. In addition to the mechanical mixing, the roughage was tamped before storage in 4 litre plastic-lidded pails. Immediately following mechanical mixing, the material was tamped or rammed with a wooden tamper or paddle. All the roughage was tamped in a large plastic mortar tray for 15 minutes. The roughage was tamped for 5 minutes for every 6 cm of material added into the pail. The value of this impact is to increase the overall lime-sand contact. Traditional techniques reveal that mortars were beaten thoroughly during mixing. The thorough mixing of the mortars affected their good adhesion properties and long-term durability. In modern free-fall mixers, the mortar is unlikely to undergo sufficient homogenization. The short mixing times may cause insufficient mixing of the lime and the sand. Tamping or beating the mixture with a wooden tamper after mechanical mixing will insure that the lime and the sand are well blended.

Two mixes of roughage were made and separately stored based on proportion of lime to sand. One type of roughage made of 1 part lime to 3

parts sand was used for Mix 1 and 2. The other type of roughage consisted of 1 part lime to 2 1/2 parts sand which was used for Mix 3, Mix 4 and Mix 5.

The roughage was stored in lidded pails with a damp piece of burlap to minimize drying. The pails were sealed and stored for one month in a shaded cool location until the addition of the brick dust or limestone dust.

Addition of Brick Dust and Limestone Dust

The correct proportions of brick dust (BD) and limestone dust (LD) were added by volume to the roughage and mixed mechanically for 15 minutes. The mortar mixtures were then tamped for 15 minutes with a wooden tamper in a plastic mortar tray. The mixtures were then added to the appropriate molds for the experimental program.

2.4.2 Molding

Molding of the samples was determined by the standard consulted for each test of the experimental program. When no standard existed, the sample size and shape was selected by the author and the supervisors of this research. The material was added to the mold in levels and tamped with a wooden paddle. Excess mortar was struck off with a trowel, and the surface was left unworked by the trowel.

Demolding of the samples occurred after 24 hours and the samples were stored on wire mesh racks in the laboratory for further curing. The

humidity and temperature in the laboratory were recorded. (See Curing Conditions, Section 2.4.3)

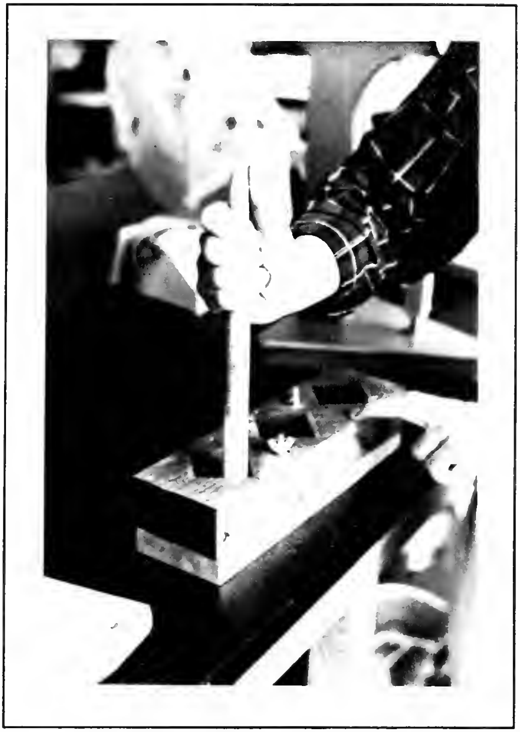

Photo 1 - Tamping with a non-absorptive, non-brittle tamper into the 50mm or 2 in Wooden Cube mold.

50 mm or 2 in Wooden Cube Molds

For the tests requiring 50 mm or 2 in cube samples, wooden three gang molds were designed and constructed following the ASTM Standard C 109 - 90. Hardwood maple molds were used as they permit water absorption

without warping on all sides of the sample, thus simulating the absorption of excess water in masonry construction.

The maple molds are held together with stainless steel hardware. They are tight fitting yet come apart to facilitate removal of the sample. Before the addition of the mortar, the wooden molds were soaked in water and toweled off to remove surface water. Soaking was done to control the rate of absorption from the setting mortar sample to the wooden mold. This in certain respects mimics the masonry practice of soaking the bricks before building with lime-based mortars. Wooden molds, rather than standard steel molds used for cement mortars, were selected for the lime-based samples in order to absorb water during the critical early period of setting.

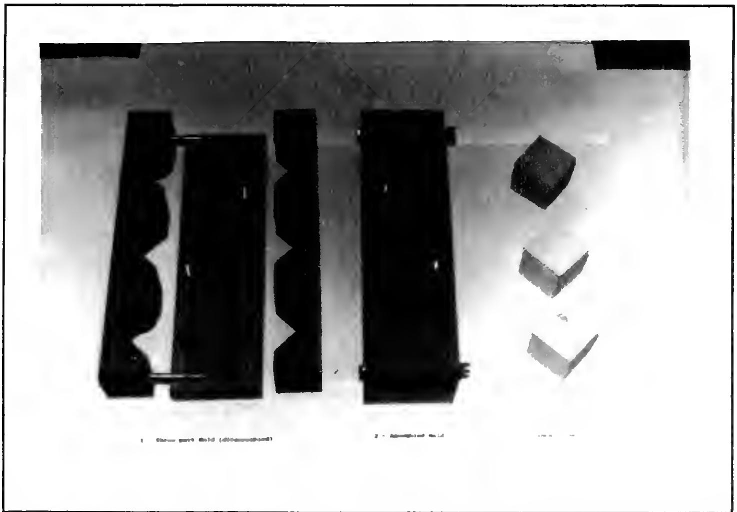

Photo 2 - Three part wooden cube mold, disassembled and assembled with 50 mm or 2 in sample cubes.

Plastic 2 3/4 in diameter by 3/4 in high Ring Molds

The molds used are rings made of rigid PVC with an interior diameter of 2 3/4 in and a height of 3/4 in high. The molds were rigid enough to prevent deformation, yet permitted removal of the mortar before full cure. The interior of the rings were sprayed with WD 40, a releasing agent. The rings were placed on porous brick in order to absorb water during the setting of the lime based material. The mortar was tamped into the mold with a wooden tamper. The brick was first soaked in water to reduce immediate absorption of water from the mortar. Prior to placing the sample on the brick, excess water was removed with a towel.

Photo 3 - Sample being demolded from PVC ring mold.

25 cm or 10 in Prism Molds

For measuring shrinkage, molds conforming to ASTM C 490 were used. These molds have two compartments containing 1 in by 1 in by 11 1/4 in prisms having a 10 in. gage length. The parts of the mold are tight-fitting and firmly held together when assembled, and their surfaces are smooth and free of pits. The molds are constructed of steel, and the sides are sufficiently rigid to prevent spreading or warping. A releasing agent was applied to the steel surfaces of the mold.

Conical Ring

To quantify setting time, a conical ring conforming to ASTM C191-82 was used. The mortar was tamped into a conical ring, resting on a glass plate about 100 mm square. The ring is made of plastic, nonabsorbent material, with an inside diameter of 70 mm at the base and 60 mm at the top and a height of 40 mm.

Table 3 - Schedule of Molds and Number of Samples

Test	Mold shape and size	# of samples
setting rate	conical ring, 70 mm/60 mm by 40 mm	1
water content	50 mm or 2 in cube	3
set under water	50 mm or 2 in cube	2
workability	conical ring, 70mm/60mm by 40 mm	2
shrinkage	1 in by 1 in by 11 1/4 in prisms	2
bulk specific grav.	50 mm or 2 in cube	2
comp. strength	50 mm or 2 in cube	4
H_2O vapor trans.	2 3/4 in diameter and 3/4 in high ring	4
depth of carbon..	50 mm or 2 in cube	3
salt resistance 1	2 3/4 in diameter and 3/4 in high ring	4
salt resistance 2	50 mm or 2 in cube	1
water absorption	50 mm or 2 in cube	2

2.4.3 Curing Conditions

As curing conditions have a significant impact on the properties of lime-based mortars, an attempt was made to achieve appropriate and overall consistent conditions for the batches. All samples were demolded after 24 hours. The demolded samples were cured on wire mesh racks in the Architectural Conservation Laboratory. The laboratory is not a climatically controlled environment and temperature and humidity fluctuated according to exterior conditions. The range of atmospheric conditions was recorded from 12.5°C to 24°C or 40°F to 75°F and 45% RH to 78% RH. This method of curing was selected as it roughly replicates exterior curing conditions, like a sheltered masonry construction. Other experimental programs have selected curing methods involving controlling temperature and relative humity.[107]

[107]Teutonico, "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars," 37.

An environmentally controlled space was not available for this experimental program. However, all the samples were subjected to the same environmental conditions, thus, a level of consistency amongst the samples was achieved.

2.5 Experimental Program Standards

When possible, testing standards were used to provide a methodology for the experimental program. North American standards were consulted for the experimental program as the materials being evaluated were North American and one of the objectives of this research was to assess the appropriateness of the standards. Some British (BRI) and Italian (NORMAL) Standards were consulted when ASTM standards did not exist. Generally, ASTM standards are geared to evaluating cement-based mortars and grouts that behave differently in terms of setting, strength development, and shrinkage.

For certain tests in the experimental program, no standards exist and thus test methods were created and closely followed. The following table includes North American Standards consulted for the experimental program. Corresponding European standards are included when known.

Table 4 - Standards Consulted for Experimental Program with corresponding European Standards

	Standard Used	Related Standard
terminology/definitions	ASTM C 51-90	
preparation of samples	ASTM C 305-82 (Reapproved 1987)	DIN 1053
color	ASTM E284	
water content	no standard	
flow	ASTM C 110	DIN 18555
setting rate	ASTM C807	
set under water	no standard	
shrinkage	ASTM C 490-89	
bulk specific gravity	ASTM C 97	
compressive strength	ASTM C109	Uni 7102 (03.06 1968)
water vapor transmission	ASTM E96	Normal, 7/81
durability as defined resistance to salt attack	BRE, Salt Crystallization Test, 1992	
water absorption coefficent	Normal 7/81	Rilem 25-Pem
microstructure	no standard	
surface morphology	ASTM 856	
pore size distribution	no standard	Normal 4/80

2.6 Experimental Program

The characterization of mortar performance requires a variety of tests to determine physical, mechanical and chemical properties. Therefore many different tests had to be reviewed and selected before the experimental program commenced. After several consultations with experts in the field of materials analysis and architectural conservation, a set a tests that would yield information on the properties of lime-based mortars modified with brick dust was selected.[108] The overall criteria shaping the experimental program was that the tests had to be reproducible in another laboratory.

Many of the selected standardized tests were borrowed from the cement industry, as few tests or testing programs have been developed specifically for lime and brick dust mortars. The testing of brick dust as a pozzolan is complex since it has no cementing properties itself and only develops in the presence of another material. Thus, it is the combination of lime and brick dust which was quantified and characterized. As a result the experimental program produced both comparative values and subjective interpretations about the materials, the mortar mixes and the test methods.

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[108]Several meetings in Fall, 1992 and Spring 1993 were held with Professors Jean Marie Teutonico and Frank G. Matero to select tests for the evaluation of the samples. Most of the tests are the same as those conducted in Phase 1 of the Smeaton Project, however the test standards differ slightly. The test Set under Water was derived from the experiments of Vicat. Examinations such as as pore size distribution, microstructure and surface morphology were not included in Phase 1 of the Smeaton project but were included in the research conducted by G. Baronio and L. Binda, "Survey of the Brick Binder Adhesion in Powdered Brick Mortars and Plasters," *Masonry International*, Vol 2, No. 3, 1988, and research conducted by the cement industry on pozzolanic additives such as A. Al-Manaseer, M. Haug and L. Wong, "Microstructure of Cement-Based Grouts Containing Fly Ash and Brine," Proceedings of the Conference on Cement, 1992, Istanbul, 635-654..

The mortar mixes were tested on their own without masonry unit such as brick or stone. Tests such as adhesion and unit strength were not conducted, but would yield valuable information if they were performed.

The experimental program was influenced by time constraints; therefore the curing time of the samples was relatively short considering the nature of this material. It would have been preferable to evaluate the mortar mixes repeatedly over a longer curing period. In this research program, some conclusions about the hardened material were made after three months curing which, given the nature of lime-based mortars, could be premature. In related research programs, the curing time was considerably longer. Researchers, investigating the mechanical properties of mortar and grout modified with natural pozzolana, brick powder and crushed bricks, performed tests after a four year curing period.[109] Historically, Vicat made observations on mortar after many years of curing. However, many mix samples will be maintained for further investigations. If a longer testing period had been followed, perhaps the results would be considerably different.

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[109] Maria Karaveziroglou-Weber and Ioanna Papyianni, "Long-Term Strength of Mortars and Grouts Used in Interventions," *Structural Preservation of the Architectural Heritage*, IABSE Symposium, Rome, 1993, 527. This research was aimed at evaluating the long-term strength development of lime-pozzolana mortars as pozzolanic reaction follows a slow and time dependent process.

The following is a schedule of the tests selected to evaluate the properties and characteristics of the mortar mixes.

Fresh Mortar	Setting Time
	Water Content
	Workability
	Set under Water
Hardened Mortar	Color/Appearance
	Bulk Specific Gravity
	Water Absorption Coefficent
	Shrinkage
	Compressive Strength
	Water Vapor Transmission
	Depth of Carbonation
	Durability - resistance to damage by salt
	Microcracking
	Microstructure
	Pore Size Distribution

2.6.1 Color

Natural pozzolanas can be found in a great variety of colors, white, black, yellow, gray, brown, red and violet. Those from Naples are predominately red, much resembling brick or tile dust.[110] Tetin from the Azores is reddish in color due to the high presence of ferric oxide.[111] Trass from Audernach Germany, (used by Smeaton at Eddystone) is yellowish gray in color.[112]

The addition of brick dust to lime-based mortar will impart color to the mixture, which should be considered before use in a masonry program. The amount, particle size, and water content are factors which will effect coloring of the mortar. Historically, it was noted that the use of coarsely pounded fragments of bricks found in the lime matrix of the Roman hydraulic works resembled a breccia.[113] Of the use of brick dust as a pozzolanic additive in the 18th century, Vicat said that the brick could not be broken up without leaving a small quantity of rather fine powder that when mixed with lime tinged slightly red or yellow, according to the color of the brick used.[114]

Although the color of the materials and mixes was not pertinent to this experimental program, it is important to note that the addition of brick dust

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110 J. G. Totten, *Essays on Hydraulic and common mortars.and on limeburning*, translated from General Treussart, M. Petot, and M. Courtois, (Philadlephia, Franklin Institute, 1842), 53.
111 Alfred Denys Cowper, *Lime and Lime Mortars,* (London, His Majesties Stationary Office, 1927), 47.
112 Ibid., 48.
113 L. J. Vicat, *A Practical Treastise on Calcareous Mortars and Cements, Artificial and Natural,* (Translated by Captain J. Smith, London, J. Weale, 1837), 119.
114 Ibid.

changes the color of the mixture. Munsell Soil Color Charts were consulted to determine the colors of the materials and the mortar.

Results

Table 5 - Color of Materials and Mortar Mixes

Brick Dust

Brick Dust 1 (Martin Clay Products)	Brick Dust 2 (Colonial Williamsburg)
2.5 YR 5/8	5 YR 6/8

Sand

Yellow Bar Sand from Dunrite Aggregates Co.	
wet - 10 YR 6/6	dry - 2.5 Y 7/6

Mortar Mixes

Mix 1	Mix 2	Mix 3	Mix 4[115]	5
2.5 Y 8/4	5 YR 7/3	5 YR 7/3	7.5 YR 7/0	7.5 YR 7/6

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[115] Mix 4, Limestone Dust, Sand and Lime was included in this analysis, although neither the additive nor the mortar is considered pozzolanic and thus resultant colour is not a consideration for use.

2.6.2 Initial Water Content

For the preparation of lime-based mortars made from lime putty, the standard practice is not to add water during the mixing process but to ram or beat the mortar until a plastic consistency is achieved[116]. Lime putty, if prepared properly, has a consistency somewhat like yogurt. It possesses enough water to be mixed with sand and other additives to yield what would be considered in the field a workable mortar. The two types of brick dust and the limestone dust were added to the mix in a dry state and no water was added to the mix. All mixes were mechanically mixed for the same period of time and then stored in lidded containers. All the mixes were handled in the same manner in an attempt to a achieve a certain level of consistency.

The amount of water in the mixture is an important factor as it may affect how the mixes will perform on tests like flow and shrinkage. As well, water is the other critical component which influences the chemical reaction of lime and pozzolana. This test serves to provide basic information on the mixes but in itself is not comparative as different mixes require different amounts of water for their optimum performance.

Standard Test Consulted - No test standard addresses this examination.

Scope of Test - The water content is determined weighing the freshly mixed sample. After drying the mortar at 80 °C for 24 hours, the sample is weighed

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[116]John and Nicloa Ashurst, *Practical Building Conservation, Vol. 3, Mortars Plasters and Renders*, (New York, Halstead Press), 4.

again. The water content of the mortar is calculated, in % by weight, from the difference in weight of the wet and dried mortar mass.

Apparatus - 1) Oven, 2) scale.

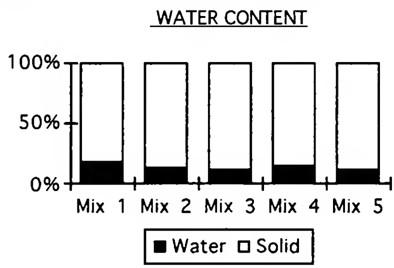
Method - A representative amount of the wet mortar mixture was weighed and placed in the oven at @ 80°C and dried for 24 hours. At the end of the drying period the weight was measured and the percent water content calculated.

Results

Table 6 : Mean Water Content of Mixes

Mix	Wet Weight (g)	Dry Weight (g)	Water Content (%)
1	169.0	138.4	18%
2	197.3	173.6	13%
3	420.1	370.2	12%
4	285.3	241.6	15%
5	354.6	312.1	12%

Graph 1- Mean Water Content of Mixes



Discussion of Results

The mean water content was calculated for each mix three times. All the mixes fell within a 3% difference in water content. The water content of Mix 1, lime and sand, was determined to be 18% of the total weight. As brick dust and limestone dust were added, the water content of the mixture was reduced. This is due to the fact that the brick dust and limestone dust were added in a dry state. Mixes 2, 3, and 5 contained brick dust and had similar water content percentages.

In future investigations, the brick dust and limestone dust, or other porous particulates, should be added in a slurry form. This would reduce the amount of water or moisture absorbed from the lime putty and result in a more consistent rate of water content and better distribution in mixing amongst the mixes being examined.

2.6.3 Workability as measured by a Flow Table

The term workability refers to the ease of spreading on an absorbent surface or the suitability for handling on a mason's tools. Workability is linked to the term plasticity, which is properly applied to clay.[117]

The workability of mortar is an important characteristic to define, as it directly affects performance and durability of the material in the field. A good lime-based mortar is evaluated on three characteristics: 1) that it adheres to the trowel or slicker without slumping; 2) that it spreads easily and; 3) that it retains water against the suction of the masonry units sufficiently to allow it time to be spread and worked it into place. Historically, attempts have been made to quantify workability of mortar. These include the Carson Blotter Test and the Emley Plasticimeter.[118] Workability of a mortar relates to consistency, thus for this experimental program it is quantified by using a flow table. This experiment does not test the properties of the mortar mixes but serves to characterize and compare them.

Standard Test Consulted- ASTM C 110 Standard Test for Flow Table.

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[117] Norman V.Knibbs and B.J. Gee, *Lime and Limestone: The Origin, Occurance, Properties, Chemistry, Analysis and Testing of Limestone, Dolomite and Their Products, and the Theory of Lime Burning and Hydration,* (Toronto, H. L. Hall, 1951), 96.

[118] Both tests were created to evaluate the workability or plasticity of lime putty. The Carson Blotter Test involves the placing lime putty on a piece of blotter paper, filter pad, and spread with strokes of a spatula over the surface. The number of strokes before the putty left the surface and rooled up under the spatula is the measure of workability. The Emley plasticimeter involves an absorbent surface and a moving trowel. A porous porcelain base on turntable is rotated with the putty being spread with simulated trowel. The torque required to rotate the turntable is indicated on a scale on the Plasticimater. from Knibbs and Gee, *Lime and Limestone: The Origin, Occurance, Properties, Chemistry, Analysis and Testing of Limestone, Dolomite and Their Products, and the Theory of Lime Burning and Hydration,* 96.

Scope of Test - To test the consistency and workability of mortars as expressed by a flow table being dropped 1/2 in, 25 times in 15 seconds. The percentage change in diameter at the base, or slump, of the mortar sample is measured to determine the consistency and thus workability of the mixture. Calculations are based on two samples of each mix being evaluated. The results are expressed as a percentage change in the diameter of the base of the sample.

Apparatus - 1) Flow table conforming to ASTM C 230-90.

Results

Table 7 - Mean Percent Change as Expressed by Flow Table

	% Change
Mix 1 (lime and sand)	6.2 %
Mix 2 (lime,sand, BD1)	5.5 %
Mix 3 (lime,sand, BD1)	5.3 %
Mix 4 (lime,sand, LSD)	9.3 %
Mix 5 (lime,sand, BD2)	5.1 %

Discussion of Results

From this test, it was observed that the addition of brick dust to the lime-based mortar reduced the slump relative to unmodified lime mortar. Mixes 2, 3 and 5, (all with brick dust) did not slump as much as Mix 1, (lime alone) on the flow table. These calculations were based on the evaluation of two samples for each mixture, whereby a maximum spread of 3% existed between the samples. Mix 4 (mixed with limestone dust) had a higher percentage of slumping relative to the other samples. Mix 4 experienced cracking and loss of cohesion during the flow table test.

In terms of workability, a lower percentage of slumping indicates that the mortar is not susceptible to crumbling and falling off the trowel or slicker during application. As well, these values influence the performance of the mortar in terms of consistency and cohesion. This relates to how the mortar will set up and whether cracking either in the mortar or at the interface of the mortar and the masonry unit could exist. A lower percentage of slumping relates to the level of consistency and cohesion of the mix, and ultimately, its performance in the masonry system.

It should be noted that these percentages have probably been affected by the water content of the mixes. As observed in 2.6.2, the initial water content of the mixes was not consistent. They ranged from 12-18%. Thus, the performance of Mix 4, (lime, sand and limestone dust) may have been adversely affected because it was the "driest" mix.

Another test, purely subjective in nature was performed concurrent to this standard test. It included working and spreading the mixes in a mortar tray with a steel masonry trowel. A certain amount of mortar adhered to the trowel and the trowel was held up from the mortar tray. The mortar was evaluated based on the length of time for the mortar to fall from the trowel. Although this test is not qualitative, it did conclude that the mixes modified with brick dust tended to stay on the trowel longer. The mix modified with limestone dust was rather crumbly and quickly fell from the trowel. Again, the water content of the mixes may have affected the results of this evaluation.

2.6.4 Setting Rate

The rate of setting of mortar is a critical test to determine if the brick dust imparts an early or pozzolanic set to the lime-based mortar. If the brick dust is pozzolanic, the rate of setting should be shorter than for a pure lime and sand mortar. This technique was invented by Louis Vicat in 1837 for his experiments on natural and artificial hydraulic limes and additives. Presently, this test is an industry standard used to identify and determine the setting rate of cement and concrete.

Standard Test Consulted _ASTM Designation C 191-82 - Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle

Scope of Test - This method covers determination of the setting time of hydraulic cement by means of the Vicat needle.

Apparatus - 1) Vicat Apparatus consisting of steel needle 1 mm in diameter, 2) conical ring with a diameter of 70 mm at the base and 60 mm at the top and a height of 40 mm.

Method - Immediately after mixing of the mortar, the standard test methods were followed. The mortar was added to the conical ring with the minimum amount of additional manipulation. The top of the ring was struck with a trowel to remove excess mortar. The Vicat needle was allowed to drop and penetrate the mortar. This operation was conducted immediately after mixing and was repeated every six hours with the depth of penetration being recorded.

The Vicat needle is used to determine when the initial and final set have occurred. Initial set is considered to have occurred when the needle stops under 35 mm from the surface of the paste. When the needle shows no appreciable indentation on the surface of the specimen, final set is considered to have occurred. The depth of penetration is measured in millimeters.

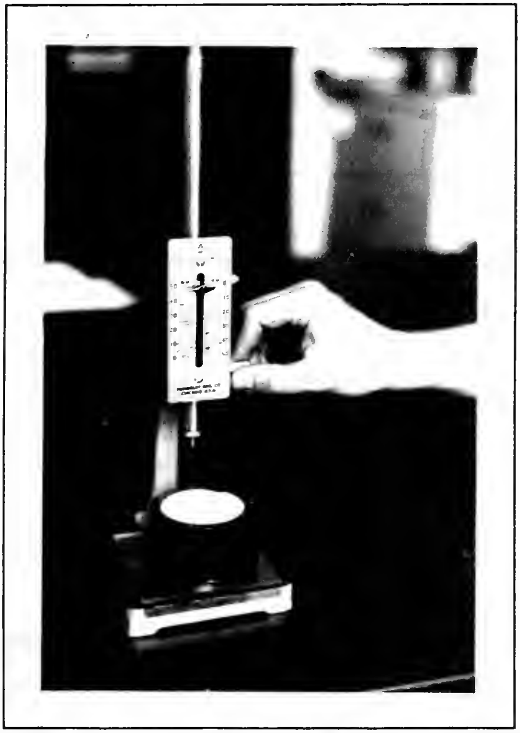


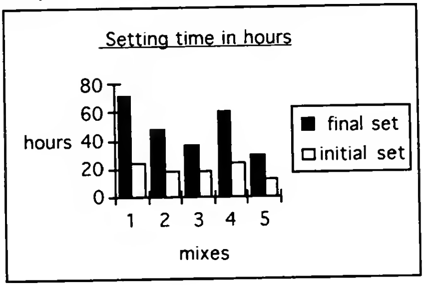
Photo 4 - Vicat Penetrometer measuring setting rate of mortar mix.

Results

Table 8 - Mean Setting Rate of Mortar Mixes - mm penetration

Time	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
0 h	0	0	0	0	0
6 h	0	0	0	0	0
12 h	0	27	29	0	35.7
18 h	24	32	31.7	30.7	45.3
24 h	37.7	41.7	45.7	41.3	48.3
30 h	41.7	46.3	48.7	45.3	50
36 h	45.3	48.7	50	48	
48 h	47.7	50		49	
60 h	47.7			50	
72 h	49.7				

Graph 2 - Mean Setting Time of Mortar Mixes



Discussion of Results

Mix 1, (lime and sand) had the slowest final setting rate of all the mixes at 72 hours. An initial set occurred after 24 hours, again the slowest initial set of all the mixes. Mix 1 serves as a control from which the other samples can be evaluated in terms of the impact of the brick dust affecting setting rate.

Mix 5 (lime, sand, brick dust 2) had the most rapid setting time at 30 hours. This mix also achieved the most rapid initial set after 12 hours. If setting time can be used to identify pozzolanicity of a brick dust, then this material could be considered the most pozzolanic of the brick dusts tested.

Mixes 2 and 3 (lime sand and brick dust 1) achieved final set respectively at 48 and 36 hours. They had initial setting times between 18 and 24 hours. If compared to the setting rate of Mix 5, it would indicate that this material is slightly less pozzolanic. As well, this seems to indicate that a slight difference in proportioning of brick dust to lime appears not to effect the setting rate.

Mix 4 (lime, sand and limestone dust) set up after 60 hours. The longer setting rate may have been influenced by the limestone dust absorbing water from the mixture and thus impeding the curing of the lime. However, this inert additive did reduce the setting time of the mixes when compared to Mix 1. In terms of other properties, the early setting rates of the lime and brick dust mortars had little relationship to eventual durability or strength.

2.6.5 Set Under Water

In an attempt to establish the degree of pozzolanicity or hydraulicity of the brick dust additives, the freshly mixed samples were placed under water and observed after demolding the 24 hour old samples. After a period of 24 hours, initial set, as indicated in 2.6.4 had occurred. Although it was anticipated that the samples would not set under water, this experiment was included to make observations about its appropriateness as an evaluative tool and to observe the behavior of the mortar mixes. In historic masonry practice, lime mortars modified with brick dust were used in projects where they would get wet or submerged in water after application. In contemporary conservation practice, lime mortars modified with brick dust have been used in maritime environments.[119] Vicat described a mortar modified with brick dust or other pozzolanas with the capacity to set under water as an eminently hydraulic or pozzolanic mortar.[120] These mortars were composed of eminently rich lime and a very energetic pozzolana.

Referenced Standard - No standard exist for this test, however a test described by Vicat served as a model for the test.

Methodology - Mix 1, 2, and 5 were used in this test, as Mix 4 had no hydraulic component, and Mix 3 was made up of the same material as Mix 2. The 2 in or 50 mm cube samples were demoulded after 24 hours of setting. One day of

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119 For example, at Caeserea, Israel, architectural conservators have used lime and brick dust mortars for the conservation of archaeological ruins in a maritime environment. In a conversation with one conservator, the situation was described whereby the mortar was used at low tide, and then hours later was completely submerged in sea water. On inspection the low tide, the mortar was not damaged.

120 Vicat, *A Practical and Scientific Treastise on the Calcareous mortars and Cements, Artificial and Scientific*, 243.

curing was necessary in order to demold and move the samples to the water filled beakers. The cubes were placed under water and observed. Photographs and descriptive notes were taken after 15 minutes, 30 minutes, 1 hour, 3 hours, 6 hours, 12 hours and 24 hours.

Results

Table 9 - Observations of the Mortar Mixes Setting under Water

time	Mix 1	Mix 2	Mix 4
15 min	-bubbles, disaggregation of surface particles	-bubbles, disaggregation of surface particles	-bubbles, disaggregation of surface particles
30 min	-disaggregation of surface in flakes, quick rate	disaggregation of surface in flakes, slow rate	-disaggregation of surface in flakes, slow rate
1 hr	- disaggregation of subsurface particles	disaggregation of surface in flakes, slow rate	-disaggregation of surface in flakes, slow rate
3 hrs	- disaggregation of subsurface particles	- disaggregation of subsurface particles	- disaggregation of subsurface particles
6 hrs	- disaggregation of inner core of cube sample	-disaggregation of subsurface particles	-disaggregation of subsurface particles
12 hrs	-complete disaggregation of cube, slump in beaker	-disaggregation of inner core of cube sample	-disaggregation of inner core of cube sample
24 hrs	-complete disaggregation of cube, slump in beaker	-disaggregation of all but inner core of cube	-disaggregation of all but inner core of cube

Discussion of Results

By placing the curing samples into water, a great deal is learned about the mortar mixes and the validity of this test. After 30 minutes mix 1 was viewed as disaggregating at a quicker rate than mixes 2 and 4. After 6 hours, the inner core of mix 1 had begun to disaggregate, while mixes 2 and 4 were disaggregating around the periphery of the still discernible cube. Although

the brick dust modified mortars were marginally more resistant to disaggregation than those without, no mix could be described as eminently hydraulic.

Although this test yielded some information regarding the hydraulicity or pozzolanicity of the mixes with brick dust relative to the unmodified lime mortars, the short period of time, 24 hours in the molds, before submerging the samples was not long enough for the samples to achieve an initial set. This test may have provided further information on water resistance if the sample had been submerged after a longer curing period.

2.6.6 Shrinkage

Shrinkage is a fundamental characteristic of mortar which affects performance and longevity of the masonry system. As a mortar sets, it will experience a certain amount of shrinkage, resulting in some internal microcracking or cracking at the interface of the mortar and the masonry unit. However, too much shrinkage is detrimental to the masonry system. The addition of pozzolana to lime based mortars has been documented as assisting in the control of shrinkage as a stronger matrix is established by the chemical bonding of the pozzolana and the lime.[121] This test attempts to evaluate the role of brick dust in controlling shrinkage of lime mortars.

Referenced Standard - ASTM C 490 -89 Standard Practice for the Determination of Length Change of Hardened Cement, Mortar and Concrete.

Scope - This practice covers the requirements for the apparatus and equipment used to prepare specimens for the determination of length change in hardened mortar, the apparatus and equipment used for the determination of these changes and the procedures for use. **Length change** is defined as an increase or decrease in the linear dimension of a test specimen, measured along the longitudinal axis, due to causes other than applied load.

Apparatus - 1) Two molds consisting of two compartments to form 1 in by 1 in by 11 1/4 in prisms. 2) A length comparator for determining the length change of samples, with a dial micrometer graduated to read 0.0001 in units.

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121 J. G. Totten, *Essays on Hydraulic and common mortars and on limeburning, translated from General Treussart, M. Petot and M. Courtois,* (Philadelphia, Franklin Institute, 1842), 140.

Calculations - The length change is calculated at any age as follows:

$L=[(L_x - L_i) \div G] \times 100$
L=change in length at x age, %
L_x= comparator reading of specimen at x age minus comparator reading of reference bar at x age; in inches
L_i= initial comparator reading of specimen minus comparator reading of reference bar at that same time, in inches
G= nominal gage length, (10).

The length change values for each specimen are calculated to the nearest 0.001% and report findings to the nearest 0.01%

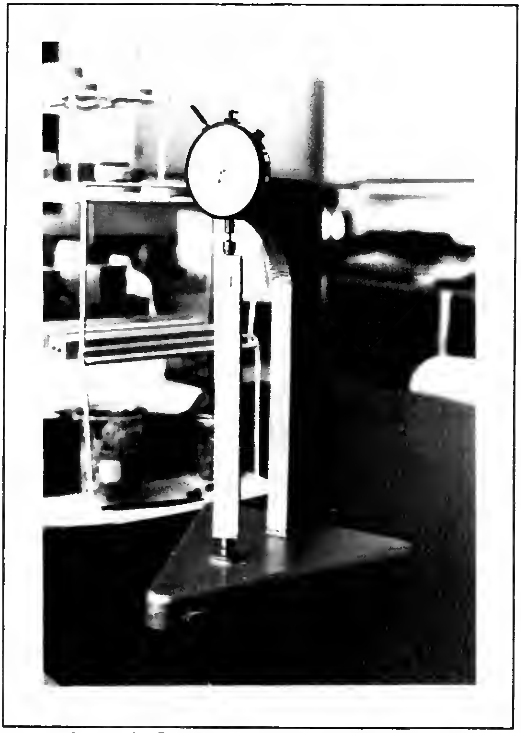

Photo 5 - Length Comparator measuring shrinkage of the prism mold.

Results

Table 10 - Mean Percent Change of Length of Mixes

Day/Hour	1.1	1.2	2.1	2.2	3.1	3.2	4.1	4.2	5.1	5.2
Day 1 - 24 h	0	0	0	0	0	0	0	0	0	0
Day 2 - 36 h	0.01	0.0004	0.002	0.002	0.007	0.001	0.005	0.005	0.001	0.003
- 48 h	0.013	0.003	0.005	0.005	0.009	0.004	0.015	0.016	0.005	0.009
Day 3 - 60 h	0.013	0.008	0.007	0.008	0.01	0.012	0.025	0.024	0.011	0.011
- 72 h	0.015	0.012	0.008	0.011	0.01	0.015	0.034	0.034	0.014	0.014
Day 4 - 84 h	0.016	0.013	0.011	0.012	0.012	0.017	0.039	0.038	0.015	0.015
- 96 h	0.016	0.013	0.014	0.013	0.014	0.018	0.042	0.042	0.016	0.016
Day 5-108h	0.018	0.014	0.017	0.014	0.015	0.02	0.044	0.045	0.018	0.017
-120h	0.02	0.016	0.023	0.017	0.017	0.021	0.046	0.045	0.019	0.017
Day 6-132h	0.022	0.016	0.023	0.018	0.018	0.021	0.053	0.049	0.02	0.018
-144h	0.022	0.017	0.024	0.019	0.019	0.022	0.053	0.051	0.02	0.018
Day 7-156h	0.023	0.017	0.024	0.02	0.019	0.022	0.056	0.055	0.02	0.019
-168h	0.024	0.022	0.025	0.022	0.019	0.023	0.059	0.057	0.022	0.02

Discussion of Results

In terms of shrinkage, Mix 1, 2, 3 and 5 displayed marginal differences after a seven day period. The addition of brick dust to the lime-based mortar decreased shrinkage negligibly, by 0.001% or 0.0001 ins over 10 in bar. From these results it appears that the addition of brick dust does not appear to affect shrinkage within a seven day curing period. Perhaps the brick dust does play a role in shrinkage after a seven day period, but this experiment did not address this factor.

In comparing Mix 1 to Mix 4, there does not appear to be any appreciable difference in shrinkage in the eary period of the experiment, however, by day seven, a slight difference 0.035% or 0.0035 ins over 10 in bar

did exist. The relative increase in shrinkage of Mix 4 compared to the other mixes could be a factor of its low initial water content or the addition of an inert particle that does not contribute to the formation of the crystalline matrix. Further investigations into the miccrostructure of the mixes can be found in 2.6.13.

Although siginificant differences were not observed for shrinkage the behaviour of the mixes, this experiment serves to evaluate the appropriateness of this standard test for lime-based materials. Essentially, this standard test is intended for cement-based materials that tend to harden and develop strength at a faster rate than lime-based materials. This standard test is not appropriate to measure the rate of skrinkage of these samples because of the slower hardening and strength development rates of lime-based materials. The size of the sample being tested (1 by 1 by 11 1/4 in prisms) does not lend itself to lime-based materials, as they are susceptible to breaking during demolding and measuring. As lime-based materials tend to harden over a longer period of time than cement-based mortars, measuring the daily rate of skrinkage over a seven day period is not long enough to evaluate skrinkage rates of the mixes.

2.6.7 BULK SPECIFIC GRAVITY

The bulk specific gravity of a material is a property that in itself is not fundamental, but influences other properties such as compressive strength, water absorption and durability. The bulk specific gravity of a material relates to its real density. Real density relates to the real, or impermeable volume of the sample. This value informs the porosity of each mix as it relates to the space of the 50 mm or 2 in cube that is permeable to water.

Scope - These test methods cover the tests for determining the bulk specific gravity. Bulk Specific Gravity is defined as the mass of a given volume of a substance divided by the mass of the same volume of water.[122]

Standard Consulted - Standard Chemistry Procedure

Bulk Specific Gravity = A/(B-C)
A=weight of the dried specimen
B=weight of the soaked and surface dried in air
C=weight of the soaked specimen in water

Apparatus - 1) scale.

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122 Shugar, Gershon J. and Ballinger, Jack P. *Chemical Technician's Ready Reference Handbook*, Magraw-Hill, New York, 3rd, 1990, 396.

Results

Table 11 - Specific Gravity of Mortar Mixes - kg/m^3

Mix	Specific Gravity
1 (lime & sand)	1.74
2 (lime, sand & BD1)	1.84
3 (lime, sand & BD1)	1.86
4 (lime, sand & LSD)	1.94
5 (lime, sand & BD2)	1.86

Discussion of Results

Not surprisingly, the addition of brick dust and limestone dust to a 2 in or 50 mm cube of lime and sand mortar does change the specific gravity of the mix. In the mixes modified with additives, more material occupies the same volume, thus increasing the bulk specific gravity. These results are not intended to be comparative, but contribute to the understanding of the behviour of these samples in other tests such as porosity, compressive strength, water vapor transmission, and resistance to salt attack.

In comparing these results to published research conducted on pozzolanic lime-based mortars, the density of the samples prepared by Pennelis et al, (1 part lime, 1 part pozzolana (Santorin), to 6 part sand) was reported as 1.86 kg/m^3 and (1 part lime, 1 part pozzolana (Santorin), to 3 parts sand and 3 parts crushed brick) to be 1.84 kg/m^3.[123] As well, these results are comparable to research conducted on pozzolanic lime mortars in Tanzania which determined the density of mixtures with varying proportions of

[123] G. Penelis., J. Papayianni, M. Karaveziroglou, "Pozzolanic Mortars for Repair of Masonry Structures," *Structural Repair and Maintenance of Historic Buildings*, 165.

pozzolanas falling between 1.72 and 1.81 kg/m^3.[124] The similar results indicate that the proportioning and mixing of the samples reflects some contemporary practice standards.

In comparing the bulk specific gravity of lime and brick dust mortars to that of Portland Cement, calculated as 3.5 kg/m^3, and hydraulic cement calculated at 3.15 kg/m^3, the porosity of these materials can be roughly compared and they are significantly more dense.[125] These differences in bulk specific gravity relate to the differences in other physical and mechanical properties between these materials.

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124 P. Cappelen, *Pozzolanas and Pozzolime,* (Dar as Salaam, Building Research Unit, 1978), 22.
125 Popovics, p. 183

2.6.8 Compressive Strength

The compressive strength evaluation of a lime-based mortar modified with brick dust proves problematic because a pozzolonic reaction follows a slow and time-dependent process. Unlike Portland Cement based mortars which achieve maximum strength in approximately 28 days, research indicates that a lime-brick dust mortar takes longer to develop strength potential. Research on the effect of particle size and on determining the reactivity of a pozzolana reports that the lime-pozzolana mortars gain strength more slowly, and reach a considerably lower ultimate strength than Portland Cement mortars.[126] For this experimental program, compressive strength evaluation was conducted at the end of the experimental program to permit the mortars a longer curing time. The mortar mixes were tested after approximately four months curing.

Compressive strength testing was included in order to further compare the brick dusts being evaluated and not as an indicator of overall final performance or durability of the mortar. The mortar was evaluated as an isolated material and not in a masonry system as it would be expected to perform.

Presently, the construction industry, including those involved in the repair of historic masonry structures, tend to think purely about compressive strength when selecting a repair mortar. As a result, mortar tends to be

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[126] P. Cappelan, *Pozzolanas and Pozzolime*, (Dar as Salaam, Building Research Unit, 1978), 7. and ASTM STP 99, *Symposium on the Use of Pozzolanic Material in Mortar and Concrete*, (Philadelphia, 1949). It has been determined that a mixture of 60% Port Cement and 40% Pozzolana has a strength equal to 75% of pure Portland Cement after 6 months, 95% after one year and 102% after 5 years.

evaluated and specified based on strength. But in many applications, high strength is not a desired property of a masonry mortar. Other characterisitics such as flexibility, adhesion and permeability are important factors for a mortar to perform its role of integrating the masonry units. Although strength of mortar can not be completely overlooked for reasons of safety, it is only one of many more important factors in good masonry.

Standard Consulted - ASTM C109-90 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or 50-mm Cube Specimens)

Scope - This test method covers determination of the compressive strength of hydraulic mortars, using 2 in. or 50-mm cube specimens.

Apparatus - 1) Specimen molds for the 2 in or 50 mm cube specimens consisted of three cube compartments. 2) The molds used were constructed of maple hardwood milled to conform to the specifications. The parts were firmly held together with stainless steel hardware. 3) An electrically driven mechanical mixer of the type equipped with paddle and mixing bowl conforming to ASTM C 305 - Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency. 4) A tamper, non-absorptive, non-brittle with a convenient length of about 5 to 6 ins. (120 - 150 mm). The tamping face shall be flat and at right angles to the length of the tamper. 5) Testing Machine: Instron Testing Machine Model 1331, stroke control (5% range) and Ramp Test Fr. 0.002 MZ.

Methodology - Three samples of each mix were selected for testing. Each sample was placed in the centre of the podium of the compression strength machine. The compressive strength machine was equipped with a sensor that detected exact moment of failure from the increased load. A computer printout for each sample was generated.

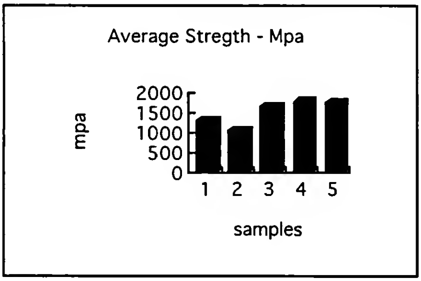
Compressive Strength testing was completed on March 25, 1994 at the Laboratory for Research on the Structure of Matter, University of Pennsylvania. Testing was conducted under the direction of Dr. Alex Radine. Data from this experiment can be found in Appendix 1.

Results -

Table 12 - Mean Compressive Strength, (Mean Mpa based on 4 cubes tested)

	Age/days	Mean Mpa.
Mix 1-Lime + Sand	141	1316.7
Mix 2- L + S + BD1	139	1055
Mix 3- L + S + BD1	134	1640
Mix 4- L + S + LSD	122	1782.5
Mix 5- L + S + BD2	119	1757.5

Graph 3 - Mean Compressive Strength of Mixes



Discussion of Results

The results of this experiment indicate that the addition of brick dust to the lime based mortars did not significantly increase the compressive strength of lime-based mortars after a four month cure. For two of the mixes 3 and 5, the compressive strength was increased marginally. However, factoring in the results of mix 2, with a lower compresive strength than lime-based mix, indicates that no overall improvement in compressive strength was achieved.

The results of this test are rather inconclusive as they are below the results reported in related research. The research completed on the calcination of natural pozzolanas reported the compressive strength of calcined opaline shale/lime mixture at 1790 Mpa, and of calcined shale/lime mortar at 1746 Mpa after a 30 day curing period.[127] In the Smeaton Project, the addition of brick dust to lime-based mortars appeared to increase the compressive strength when the particle size of the brick dust was small. Compressive strength of these mortars was reported at 2.18 N/mm^2 to 2.43 N/mm^2 or Mpa after a 120 day curing period.[128]

Generally, the addition of brick dust increased the compressive strength of the mortar samples. An explanation for this could simply be that more material is packed into the volume. As witnessed by the bulk specific

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[127] R.C. Mielenz, "Mineral Admixtures - History and Background," *Concrete International*, 1983.
[128] Teutonico, "The Smeaton Project: Factors Affecting the Properties of Lime-Based Mortars, " *APT Bulletin*, 41.

gravity values, the modified mixes are denser and thus could have higher rates of compressive strength.

Factors affecting this experiment that may have caused the inconclusive results include: 1) the rough surface of the mortar cube causing the sample to crack during compression. The compresive strength testing machine is sensitive to cracking or any movement of the sample; 2) The samples were not always placed in the compressive strength testing machine in the same orientation. Because the top trowelled surface was sometimes rough, the samples were turned on their side. This difference in placemant may have impacted this test, as tamping during the molding may have created statifications in the cubes which were susceptible to premature cracking.

For more conclusive results, this examination should be repeated. If this test was repeated, it should examine the different mortar mixes after a longer curing period and examine the development of strength at various stages of the curing of the mixes. A three point rupture test could shed light on the compressive strengths of lime-based mortars.

This test was included in the experimental program in order to give quantifiable and comparable figures to the mixes being examined. Generally, the results of compressive strength testing should not strongly inform the selection of a repair mortar. Strength of the mortar is not an important property as it is relative to the masonry unit and the masonry system. As well, high compressive strength does not equate good performance or durability in a masonry system.

2.6.9 Water Vapor Transmission

The purpose of this test is to obtain, by means of simple apparatus, reliable values of water vapor transfer through the mortar samples, expressed in suitable units. Water vapor transmission is an important quality of mortar in a masonry system as it relates to the permeability of the material to allow water vapor to exit the masonry system via the mortar joint. In some historic masonry systems, the mortar joint was generally designed to be the conduit for water and water vapor to escape. As well, water vapor transmission rates are important factors for determining compatibility of masonry systems. Therefore, depending on the nature of the masonry system, it is desirable to use a durable mortar that can transfer liquid water and water vapor. This ability to transfer liquid water and water vapor out of the masonry system reduces the susceptiblity to deterioration. Trapped liquid water or water vapor can freeze resulting in cracking of the mortar and/or masonry unit. As well, wet materials have a lower compressive strength and thus could fail under stress or load.

Standard Consulted - ASTM E 96-90 - Standard Test Methods for Water Vapor Transmission of Materials

Scope- To determine the water vapor transmission of the mortar. The methods are limited to samples not over 1/4 in. (32 mm.) in thickness. The water method was selected because it has been successfully used on mortars and paints.

In the water method, the test specimen is sealed to the open mouth of a dish containing distilled water, and the assembly placed in a controlled atmosphere with desiccant. Periodic weighing determine the rate of water vapor movement from the specimen into the desiccant.

Terminology - Water vapor permeability is defined as the time rate of water vapor transmission through unit area of flat material of unit thickness induced by unit vapor pressure difference between two specific surfaces, under specified temperature and humidity conditions.

Water vapor transmission rate is defined as the steady water vapor flow in unit time through unit area of a body, normal to specific parallel surfaces, under specific conditions of temperature and humidity at each surface.

Apparatus - 1) Scales. The scale was checked daily with a known weight. 2) The molds used for this test were rings made of rigid PVC with an interior diameter of 2 3/4" and 3/4" high The molds were rigid enough to prevent deformation, yet permitted removal of the mortar. 3) The test dish used was a tri-cornered polypropylene 250 ml. beaker. These beakers have an inner diameter of 2 3/4 ". 5) For a desiccating chamber, a glass fish tank with a glass lid fighted with a gasket was used. Three glass trays were filled with desiccant. The desiccant used was Drierite or Ca SO_4 (anhydrous calcium sulphate) size 8 mesh, manufactured in USA by W. A. Hammond Drierite Co. Xenia, Ohio. The desiccating chamber was covered with a sheet of glass. The top of the fish tank was sealed with rubber weather stripping to assist in maintaining a controlled atmosphere. 6) Two hygrometers were used, one to measure the

humidity in the tank, the other to measure the humidity of the laboratory. A second hygrometer was periodically used to verify the primary hygrometers. 7) Two thermometers, one to measure the temperature in the tank and the other for outside the tank.

Temperature and Humidity - The relative humidity in the desiccating chamber was maintained between 5 and 10 % RH and measured on a daily basis. The desiccant was changed as required as determined by changes in the relative humidity inside the tank and by the color change of the desiccant indicator. The temperature of the chamber fluctuated from 14 to 24°C. It was impossible to control the temperature of the chamber as the temperature outside of the chamber could not be controlled.

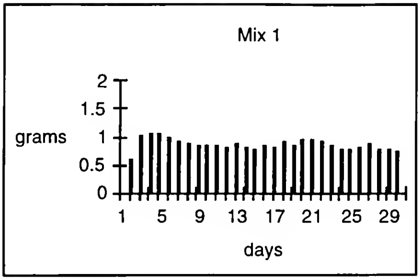
Methodology - Sample rates were measured on an electronic scale that was calibrated after each weighing by adding or subtracting the differences in the weight of known weight.

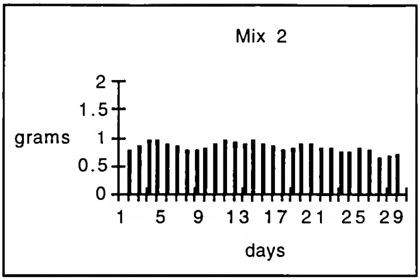
The daily rate of water loss due to water vapor transmission was calculated for each sample. The mean value for each mortar mix was calculated and graphed. Calculations were based on the mean of four samples for each mix.

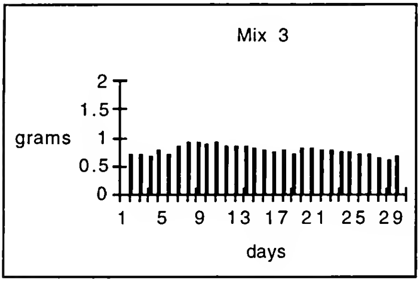
Table 13 - Mean weight change of assemblies - (g)

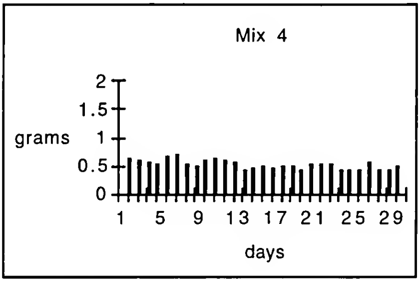
	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Day 1	0	0	0	0	0
2	0.61	0.79	0.69	0.62	0.74
3	1.04	0.85	0.71	0.6	0.8
4	1.07	0.95	0.66	0.58	0.9
5	1.06	0.97	0.76	0.52	0.84
6	0.99	0.89	0.72	0.68	0.89
7	0.91	0.86	0.84	0.71	0.94
8	0.88	0.79	0.9	0.52	0.93
9	0.86	0.78	0.92	0.49	0.85
10	0.84	0.82	0.88	0.59	0.95
11	0.83	0.87	0.91	0.64	0.93
12	0.8	0.94	0.84	0.59	0.98
13	0.87	0.93	0.83	0.55	1.01
14	0.8	0.88	0.84	0.42	0.98
15	0.79	0.95	0.81	0.47	0.94
16	0.84	0.87	0.79	0.48	0.99
17	0.81	0.86	0.75	0.46	0.97
18	0.91	0.78	0.76	0.49	0.93
19	0.86	0.82	0.69	0.49	0.85
20	0.94	0.89	0.82	0.43	0.89
21	0.94	0.88	0.81	0.53	0.87
22	0.91	0.80	0.78	0.54	0.94
23	0.85	0.82	0.77	0.52	0.95
24	0.79	0.75	0.75	0.42	0.9
25	0.77	0.75	0.73	0.44	0.91
26	0.81	0.82	0.71	0.42	0.96
27	0.89	0.78	0.71	0.56	0.93
28	0.78	0.65	0.62	0.43	0.85
29	0.76	0.67	0.61	0.41	0.87
30	0.73	0.69	0.68	0.51	0.85

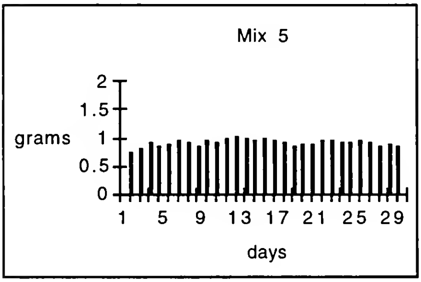
Graph 4 - Mean Water Vapor Transmission











Discussion of Results

The results indicate that a slight difference in the water vapor transmission between the some of the mixes existed. In mix 1 (lime and sand) and mixes 2 and 3, (lime, sand and brick dust 1), there was no significant difference in the WVT. The addition of this type of brick dust, irrespective of proportion, has little or no affect on the WVT of the lime-based mortar. However, the addition of brick dust 2, as witnessed by mix 5, did marginally increase the WVT of the lime-based mortar. In comparing these results to the observations made in 2.6.13, Microcracking, mix 5 had the smallest and least number of microcracks. This suggests that the addition of this type of brick dust to the lime did create a strong, yet permeable matrix.

2.6.10 Water Absorption Capacity

From this simple test, water absorption level and rate can be calculated. The rate of water absorption is measured to compare the behaviour of the lime mortar with the addition of brick dust or a porous particulate. W.A.C. results will influence other tests such as water vapor transmission and porosity.

Standard Consulted - Normal 7/81, as reported by Jeanne Marie in *A Laboratory Manual for Architectural Conservators,* Teutonico, 1988.

Methodology - Two 2 in cubes of five different mixes were washed with deionized water to remove powdered material from the surface.The samples were dried for 24 hours at 60°C. The samples were permitted to cool in a humidity controlled environment (see water vapor transmission) Intial weighing of the sample took place, recorded as M_o. The drying process was continued until the mass of the sample was constant. This was acheived after 3 cycles. The samples were placed in 500 ml glass beakers and deionized water was added until the samples were covered with 2 cm of water. The samples were weighed at regular increasing intervals; at each chosen time, the sample was taken out of the water, blotted with a paper towel, and then weighed.

Calculations - At each interval, the quantity of water absorbed with respect to the mass of the dry sample was expressed using the following calculations:

$$\Delta M/M_o \ \% = [M_n - M_o \ / \ M_o] \times 100$$

where M_n = weight of the wet sample at time t_n and M_o = weight of the dry sample.
M_o = weight of the sample after drying.

The Water Absorption Capacity was then calculated using the following calculations:

$$WAC = [M_{max} - M_d / M_d] \times 100$$

where M_{max} = the mass of the sample at maximum water absorption
M_d = the mass of the sample after redrying at the termination of the test.

Results

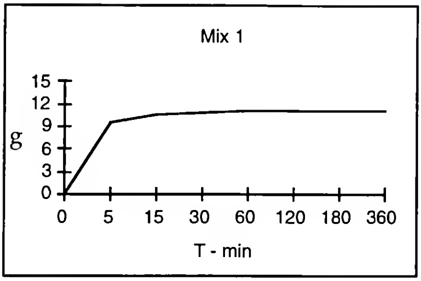
Table 14 - Mean Water Absorption of Mixes - (g)

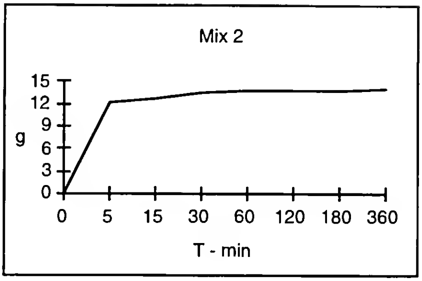
		Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
5 min	WA	9.6	12.3	10.2	9.4	11.29
15 min	WA	10.7	12.7	11.2	9.5	11.85
30 min	WA	10.8	13.6	11.8	9.8	12.1
1 hour	WA	11.0	13.7	12.0	10.1	12.3
2 hour	WA	11.0	13.8	12.2	10.3	12.4
3 hour	WA	11.0	13.8	12.3	10.3	12.5
6 hour	WA	11.1	13.9	12.3	10.4	12.5

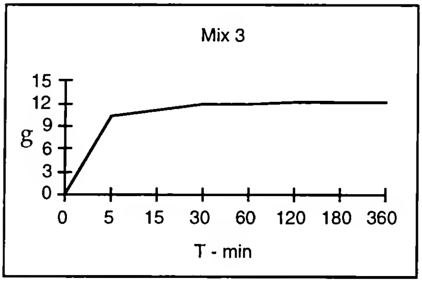
Table 15 - Mean Water Capacity of Mixes - (g)

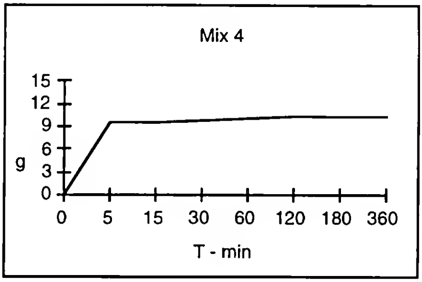
	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
WAC	10.9	13.7	12.2	10.1	12.5
acheived at	1 hr	1 hr	2 hr	1 hr	3 hr

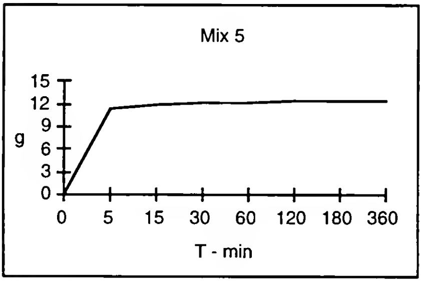
Graph 6 - Water Absorption Curve - Mean Value - (g)











Discussion of the Results

From this experiment it can be seen that the addition of both types of brick dust marginally increased the water absorption capacity of lime-based mortars. Although this experiment does not have any direct relationship to the performance of these mortars in a masonry system, it does shed light on the results of water vapor transmission rate and the liquid water permeability. In comparing the results of the two experiments, it can be said that the addition of brick dust slightly improves the ability of the samples to absorb and transfer water through the lime based mortar. However, no statements can be made regarding water effectively getting out of the masonry system.

2.6.11 Depth of Carbonation

The measuring of the depth of carbonation evaluates the long term curing rate of the sample. It should be noted that this test is not a standard, but can be effectively used to establish trends in the curing of the mixes. As all the mixes were subjected to similar curing conditions, the test serves to indicate how the constituents of the mix affect curing of the sample.

Standard Consulted - Exercise 26, "Investigation of the carbonation process in lime mortars by means of phenolphthalein," *A Laboratory Manual for Architectural Conservators*, Teutonico, 1988.

Scope

This test indicates the progress of carbonation or curing of a lime based sample through the use of phenolphthalein indicator. Phenolphthalein reacts to alkaline materials and is colorless in an acid or neutral environment. In a freshly cut or broken sample, the phenolphthalein will indicate the depth or progress of carbonation of the mortar sample by reacting to the alkalinity of the free or uncarbonated lime. This level was measured with a ruler calibrated in millimeters on three different samples (50 mm or 2 in cube) at the 30, 60 and 90 day cure. Three samples of each mix were measured to yield the average measurement of depth of carbonation.

Results

Table 16 - Mean Depth of Carbonation of Mixes - (mm)

Days	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
30	2	2.5	3	1.8	3.1
60	4.3	5.2	5.3	4.2	5.8
90	5.5	6.2	6.4	5.4	6.7

Discusion of Test Results

Though not a precise measurement, this test does indicate certain characteristics about the mixes and their constituent parts. The test indicates that the addition of brick dust does have an affect on the curing or carbonatization of lime-based mortars as exhibited by those mixes with brick dust versus the unmodified mixes. In comparing the efficacy of the brick dust additives, Mix 5 exhibited deeper levels of curing at the 30, 60 and 90 day rate than Mix 2 and 3.

It should be noted that it was difficult to achieve an accurate reading for Mix 4 as the limestone dust was affected by the phenolphthalein and marred the line or level of carbonatization. In future experimental programs the measurements should be continued for a longer curing period, as 90 days may not be long enough for lime-based materials. In comparing these results to those recorded in 2.6.4, Setting Rate, the same trends exist.

2.6.12 Resistance to Salt Attack

Historically, it was found that the addition of pozzolanas improves the resistance of lime-based materials to salt attack. Vitruvius mentioned this phenomenon and investigations of Smeaton lead to the addition of pozzolana to his lime mixture at Eddystone. In this century, the cement industry has appreciated that the addition of pozzolanas improves resistance to sulphate waters.[129]

A mortar resistant to salt attack has been a long sought after material in conservation which in part has led many to use cement-based mortars or to add cement to lime-based mortars, thus equating strength with durability. The addition of a pozzolanic material is thought to improve salt resistance because a strong yet porous and permeable matrix is established whereby salts in solution can pass through the mortar to the surface without deteriorating the material along the way. This experiment attempts to compare the durability of lime mortar to that of lime mortars modified with brick dust or porous particulate as measured by the Salt Crystallization Test.

Standard Consulted - British Research Establishment Report, Crystallization Test, 1992 (modified by the author)

The test standard was designed to be used for building stone. It was modified for this research in terms of concentration of the salt solution and a desiccator was not used.. The crystallization test involves 12 cycles of submerging the 2 in disc-shaped samples in a 10% solution of sodium sulphate by weight. The samples were dried in an oven at 100°C for 24 hours

[129] *Symposium on the Use of Pozzolanic Materials in Mortar and Concrete.*, 12.

prior to submerging. The samples were weighed and then submerged in sodium sulphate solution for 2 hours. The samples were then placed in the oven for 24 hours at 100°C. The samples were weighed and photographed. The cycle was repeated 12 times. Disc samples were used for this experiment due to availability.

During the wetting cycle, the porous and permeable samples are saturated with the salt solution. Upon drying in the oven, the liquid water is removed and soluble salts return to the solid or crystal state in the pores and on the surface of the samples. The force of this action often causes the host material to crack or diaggregate. Thus, this examination measures weight loss as a factor of resistance to salt attack.

It should be noted that a small amount of material was sometimes lost in the handling of the samples. When an appreciable amount of material was lost, this was noted.

Calculations

Calculate the mean percentage weight loss for each set of samples

% weight loss = $100 (W_f - W_1)/W_0$
W_f= weight of sample after cycle
W_1= weight of sample after label
W_0= weight of sample after oven drying

(note: there was no change in weight after label was added as a permanent felt type marker was used to identify samples)

Results

Table 17 - Mean % Weight Change of Mixes (g) - Experiment 1

Mix	1.3	1.4	2.3	2.7	3.3	3.4	4.1	4.8	5.3	5.4
weight g.	129.9	123.5	110.5	125.4	122.7	132.8	140.3	140.4	135.5	133.7
Cycle 1	+0.81	+0.83	+0.77	+0.69	+0.66	+0.79	+0.59	+0.61	+0.82	+0.75
Cycle 2	+1.46	+1.51	+1.26	+1.35	+1.35	+1.58	+1.25	+1.51	+1.68	+1.44
Cycle 3	+1.92	+2.03	+1.59	+1.77	+1.79	+2.16	+1.67	+1.98	+2.42	+2.16
Cycle 4	+2.41	+2.34	+1.77	+2.17	+2.19	+2.34	+2.53	+3.4	+3.1	+2.63
Cycle 5	+2.84	+2.74	+1.92	+2.59	+2.6	+2.81	+2.37	+2.86	+3.52	+3.29
Cycle 6	+3.13	+2.89	+1.97	+2.88	+2.75	+3.06	+2.73	+3.15	+4.12	+3.56
Cycle 7	+3.31	+3.18	+2.07	+2.95	+2.91	+3.41	+2.87	+3.37	+4.34	+3.79
Cycle 8	+2.94	+2.92	+1.73	+3.08	+2.9	+3.24	+2.76	+3.27	+0.66	+3.46
Cycle 9	+2.82	+2.89	+1.45	+3.23	+2.9	+2.88	+2.46	+3.27	+0.09	+3.18
Cycle 10	+2.69	+2.75	+1.38	+3.35	+2.72	+2.78	+2.27	+3.19	+0.07	+2.94
Cycle 11	+2.62	+2.63	+1.29	+3.12	+2.5	+2.63	+2	+2.83	-0.57	+2.71
Cycle 12	+2.57	+2.49	+1.18	+3.03	+2.35	+2.42	+1.86	+2.45	-1.49	+2.35
% change	+1.9	+2.02	+1.07	+2.41	+1.91	+1.82	+1.32	+1.74	-1.09	+1.75

Discussion of Results

Interpretation of the results of the salt resistance test indicate several trends, but also yields some inconsistencies in the test program. The samples tended to gain as opposed to lose weight. Weight gain was probably caused by the salts forming in and on the surface of the samples, without causing the samples to lose material. As well, all the samples seemed to offer some resistance to sulphate action with the addition of the brick dust not clearly impacting the lime mortar.

Preparation of the samples may have had an impact on this test, as the trowelled surfaces of the disc tended to crack after the sixth cycle. The cracks widened and surface delamination was apparent. The perimeter of the disc was susceptible to disaggregation because this area of the disc was not finished.

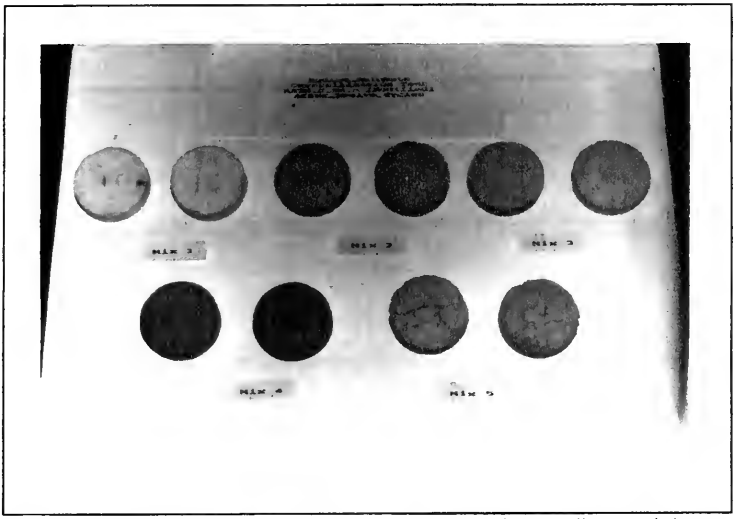


Photo 6 - Samples after 12 cycles of 10% solution sodium sulphate crystallization test

Due to these rather strange results, it was decided to conduct this experiment a second time. The concentration of the sodium sulphate solution was increased to 14%. 2 in or 50 mm cubes were used rather than discs. Only one cube of each mix was available for the experiment.

Results

Table 18 - % Weight Change of Mixes (g) - Experiment 2

Day	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
1	+1.58	+1.57	+1.56	+1.48	+1.43
2	+5.25	+5.33	+5.94	+5.42	+6.52
3	+8.84	+6.18	+9.02	+8.09	-6.76
4	+9.73	+4.76	+8.27	+9.29	-15.32
5	+9.86	+1.67	+10.88	+10.00	-31.51
6	+13.06	+6.28	+14.55	+12.42	-31.23
7	+6.58	+2.91	+14.62	+12.53	-46.28
8	+6.37	+2.65	+15.06	+12.91	-48.43
9	+2.85	+2.66	+17.18	+15.42	-56.11
1 0	-2.89	+1.65	+17.78	+2.61	-57.1

Discussion of Results of Experiment 2

In experiment 2, the increased concentration of the sodium sulphate did have dramatic effects on some of the mixes. As only one sample of each mix was used for the experiment, conclusions are tenuous. In this experiment it can not be stated that the addition of brick dust improved the resistance of the samples to salt attack. The mixes modified with brick dust behaved significantly different. Mix 2 exhibited a slight increase in weight. Mix 3 exhibited a significant increase in weight. While mix 5 exhibited a very significant decrease in weight. Mixes 2 and 3 exhibited cracking at or near the tenth cycle, while mix 5 had lost one quarter of its original mass due to cracking. Perhaps an explanation for the dramatic weight loss of mix 5 is the clay type. Further study of this material is required as other experiments suggest it behaves as a pozzolana.

The weight gain for mixes 2, 3 and 4 is attributed to the salts in solution entering the porous structure and crystallizing upon drying. Although the use of cubes rather than discs permits better observations for this experiment, this experiment makes comparison of highly porous materials difficult. In future related experimental programs other methods of durability testing should be considered.

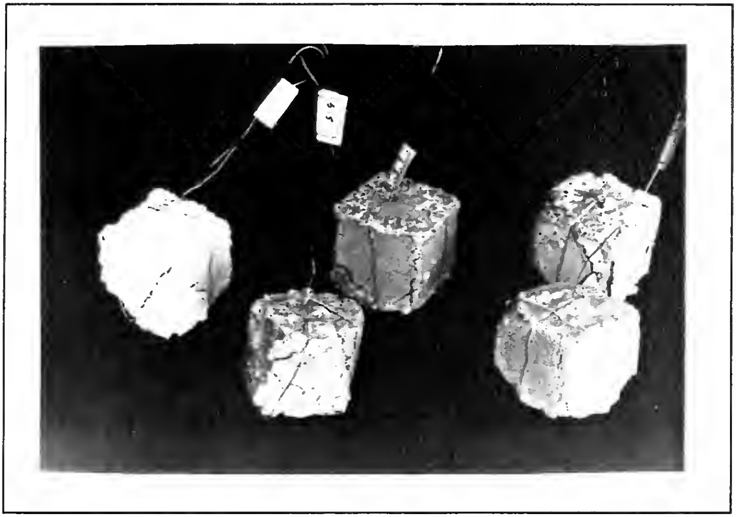


Photo 7 - Samples after 10 cycles of 14% solution sodium sulphate crystallization test, note the cracking of the cubes and salts on the surface of the cubes.

2.6.13 Microcracking of Mortar Mixes

Thin section microscopical examination of the mirostructure of mortar mixes can provide supplementary information that can not be obtained from standard physical and chemical tests. Microcracking of the mortar can be identified and quantified by using micrometry. The long term performance of the mortar is a function of microcracking as these small fissures can trap water and harmful salts. Microcracking indicates shrinkage and lack or loss of intergranular bond. Similarly, the role of the brick dust and the porous particulate can be evaluated by observing the character and location of the microcracks.

Apparatus - 1) Nikon Optiphot Polarized Light Microscope, 2) Micrometer, 3) prepared thin section of mortar samples.

Methodology - Using thin section microscopy, each mortar sample was examined in transmitted, plain and polarized light. Three representative areas were selected to make the observations and measure the size of the microcracking. The width of all the cracks in each selected area were measured and recorded. The measurements reported are the averages of the three representative areas. As the sand in the mix is 99.5% silica, (see Section 2.3) it is clearly distinguishable in polarized light. The samples were viewed in the microscope at 10 x. A photomicrograph was taken of each representative area.

Measurements - The micrometer at 10 x was calibrated at 1μm equal to 0.012 mm.

Observations

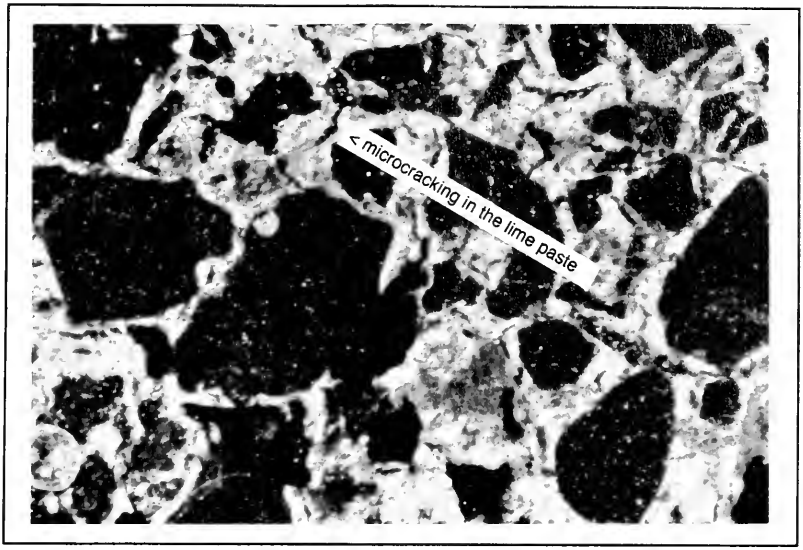


Photo 8 - Microphotograph, 10 X Mix 1

Mix 1

This mix, consisting of lime and sand, exhibits microcracking in the lime paste. No cracks were located at the interface of the paste and aggregate which suggests that the samples are well mixed and that aggregate-alkali reaction[130] is not present. The cracks measure approximately 2-5 μm or 0.024 mm to 0.06 mm wide and the length varies considerably. Microcracking in the lime paste was anticipated based on other researchers obervations.

[130] Alkali- aggregate reaction is a phenomenon associated with the use of a reactive form of silica from the aggregate reacting with certain alkaline constituents from the cement. Although most commonly associated with cement and concrete, it has been noted in lime based materials when the reactive aggregate has been used. Lea, *The Chemistry of Cement and Concrete*, 569.

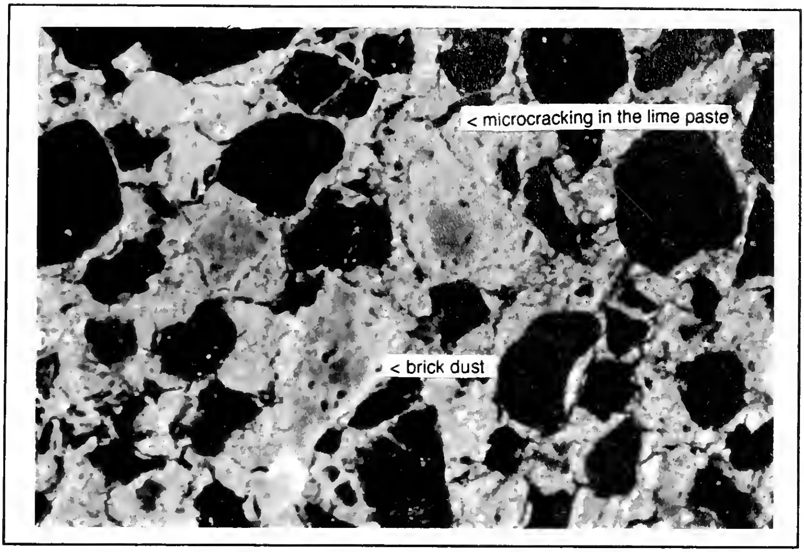


Photo 9 - Microphotograph, 10 X Mix 2

Mix 2

In this mix, brick dust and lime comprise the paste. Microcracks observed range from approximately 2 to 3 μm or 0.024 to 0.036 mm wide, less than in the lime matrix. Like Mix 1, the length of the cracks varies considerably. These cracks generally occur in the paste matrix, but some microcracks exist at the interface of the lime and brick dust and at the interface of the lime and the sand. The smaller particles of brick dust appear to be well mixed into the lime.

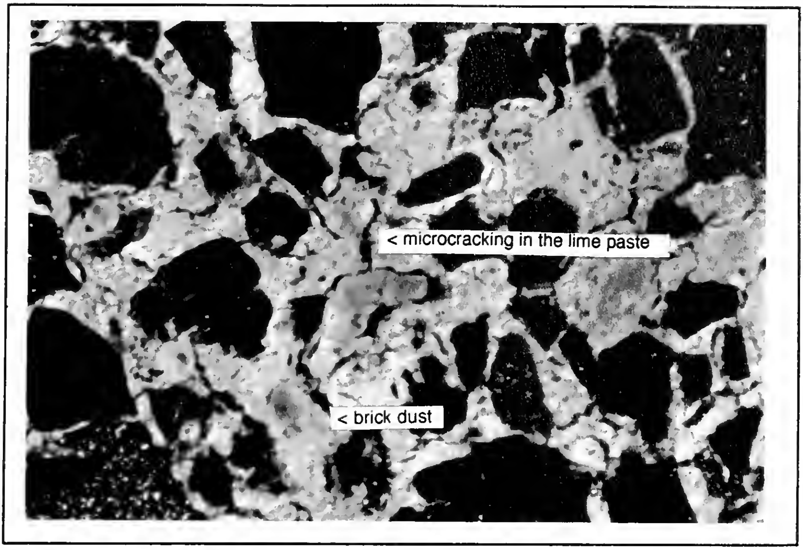


Photo 10 - Microphotograph, 10 X Mix 3

Mix 3

Microcracks in this mixture have been measured at 2-3 μm or 0.024 to 0.036 mm wide, and the length varies considerably. The microcracking generally occurs in the paste matrix between either particles of sand or brick dust. Some microcracking can be found at the interface of the lime and brick dust and the sand particles, as was found in mix 2.

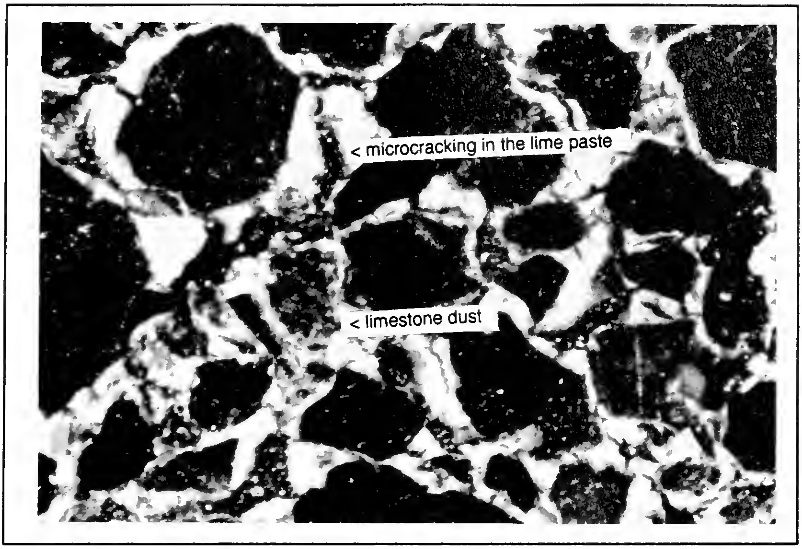


Photo 11 - Microphotograph, 10 X Mix 4

Mix 4

In this mix, lime mortar modified with limestone dust, the microcracks range in measurement at approximately 4-6 μm or 0.048 to 0.072 mm wide. These cracks are a little larger than those found in mix 1. However, these microcracks were the largest observed amongst the mixes. The cracks are both in the lime matrix and at the interface of the lime and limestone dust. The existence of microcracking around the porous particulate is expected, as no special reaction or chemical bonding appears to be formed between the lime and limestone dust. The microcracks are probably the result of the absorption of available water from the paste by the limestone dust.

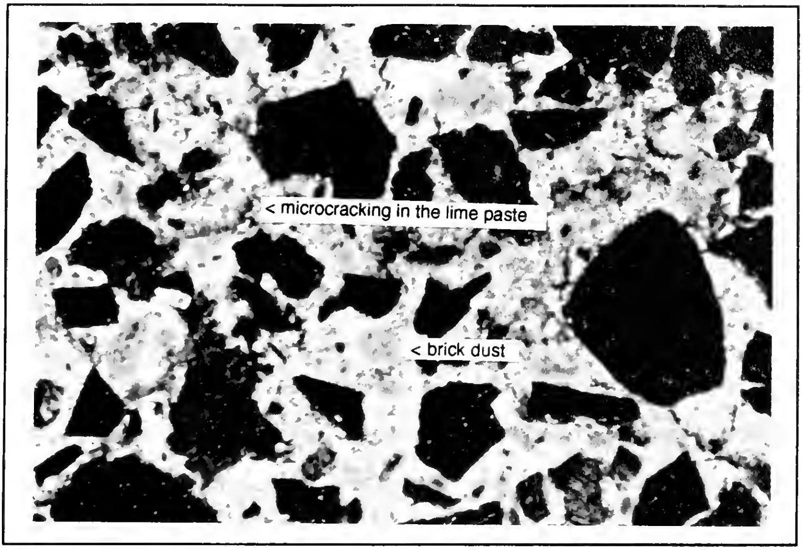


Photo 12 - Microphotograph, 10 X Mix 5

Mix 5

In this mix; lime sand and brick dust 2, microcracks measure from 1 to 3 µm or 0.012 to 0.036 mm wide and appear to be fewer than in the other samples. These microcracks appear to be in the paste matrix, and around the sand. There does not appear to be microcracking around the brick dust particles.

Discussion of Observations

The presence of microcracking in all the mixes indicates that the addition of a brick dust does not eliminate microcracks in lime-based mortars. However, the addition of brick dust does reduce the size and number of the microcracks. The addition of BD 2 to the lime-based mixes tends to reduce the size and number of microcracks more than does BD 1. The difference in proportions of BD 1 does not seem to have any effect on microcracking.

The addition of the limestone dust seems to increase the size and number of microcracks in the lime-based mixture. However, these cracks could be caused by the limestone dust absorbing available water in the mix, and thus causing microcracking in the lime. This mix was particularly dry at the time of initial curing. The same phenomenon appeared to have occurred in the setting rate of the mixes, Section 2.6.4, whereby the setting was impeded by a reduced water content.

The minimizing of microcracking in a mortar is sought after in the field, as microcracks significantly reduce durability. Microcracks permit water and deleterious solubles to enter and wick deeper in to the masonry system where they can become trapped. As well, microcracks create weaknesses in the mortar, and may result in larger cracks. The addition of brick dust to lime-based mortars does seem to reduce the size and amount of microcracking rendering a more durable cured matrix.

In relating these observations to 2.6.12, Resistance to Salt Attack, no correlations can be made. Samples with the least microcracks, mix 2, 3 and 5 had no consistent resistance to salt attack. Similarly, mix 4, had the largest microcracks but yet performed comparatively well in the salt resistance testing.

2.6.14 Microstructure of Mortar Mixes

Additional study of the microstructure of the mortar mixes involved the utilization of scanning electron microscopy to permit observation of the samples under greater magnification coupled with X-ray analysis of the constituent elements. For the purposes of this examination, SEM was used in an attempt to view the relationship of the constituents and the interface of lime and brick dust. Mapping of the constituent elements of the mixes was also conducted.

Apparatus - 1) Prepared samples of mortar mixes 2) JAOL 6400 Scanning Electron Microscope equipped with X-ray analyser.

Methodology - After a six month curing period samples of Mix 1, Mix 2, Mix 4, Mix 5 and BD 1 and BD2 were carbon-coated and observed using the SEM. Each sample was observed on the SEM and a photo taken of a representative area. Qualitative energy dispersive x-ray analysis was also conducted on each sample.

The photographs and the elemental analysis generated from the SEM are included in this report. This experiment was conducted at the Laboratory for Research on the Structure of Matter at the University of Pennsylvania on July 19, 1994 under the direction of Dr. Xue Chin Wong.

Observations

Brick Dust 1

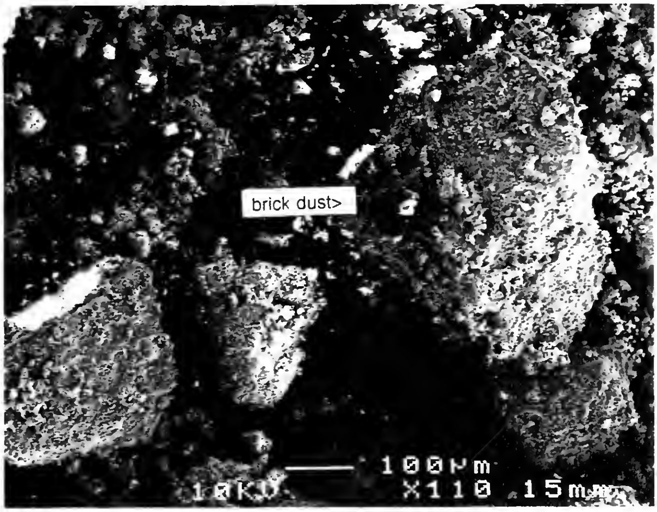
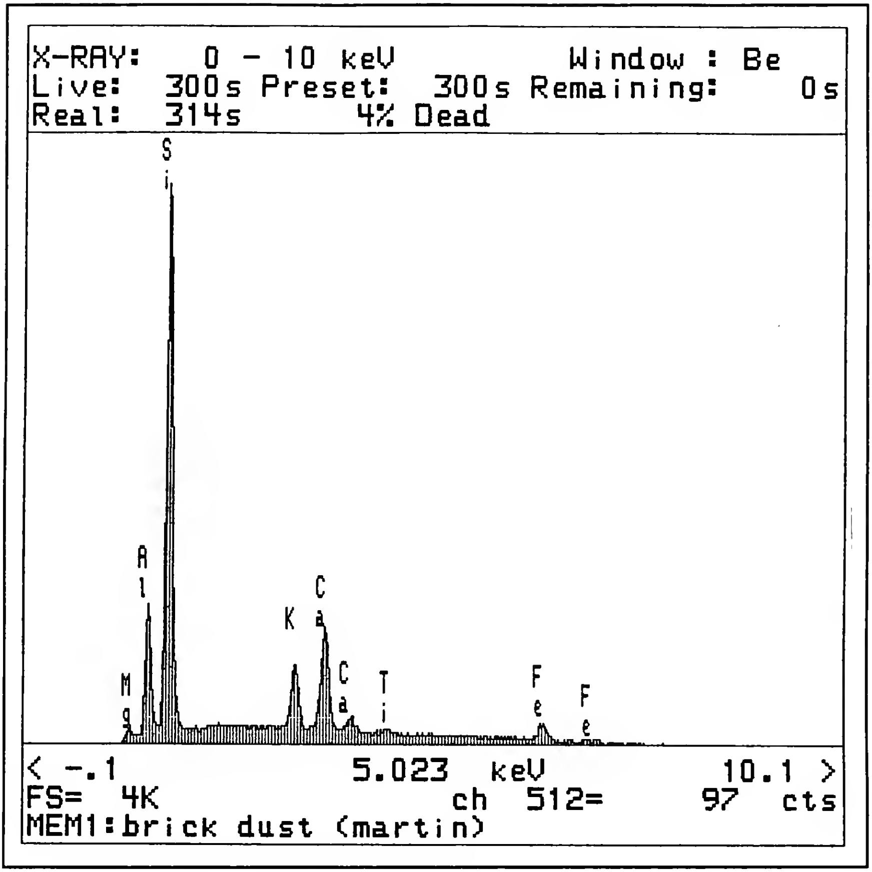


Photo 13 - SEM microphotograph, Mag. x 110, Brick Dust, (BD 1)

The particles are generally subrounded and highly porous with rough surfaces ranging from 300 µm to 75 µm (based on sieve analysis).



Graph - Elemental Spectragram, Brick Dust, BD 1

Elemental analysis of brick dust 1 indicates a high silicon content as would be expected. Other elements detected include aluminum, calcium, potassium, iron and trace amounts of titanium. The high silicon content of the brick dust suggests the presence of silica and the potential pozzolanic reactivity of the material.

Brick Dust 2

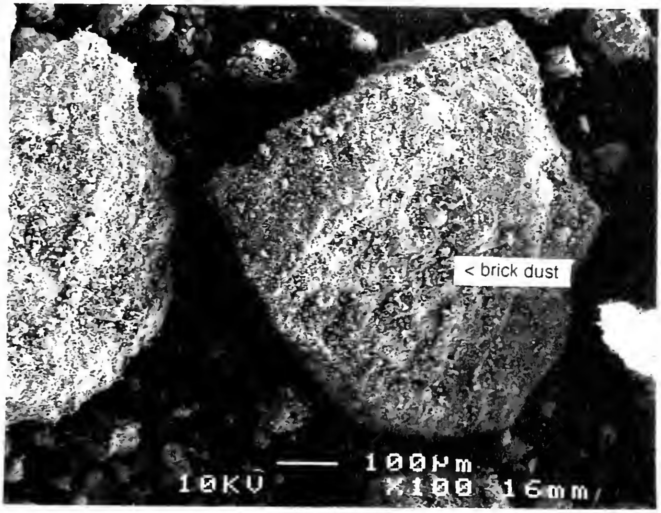
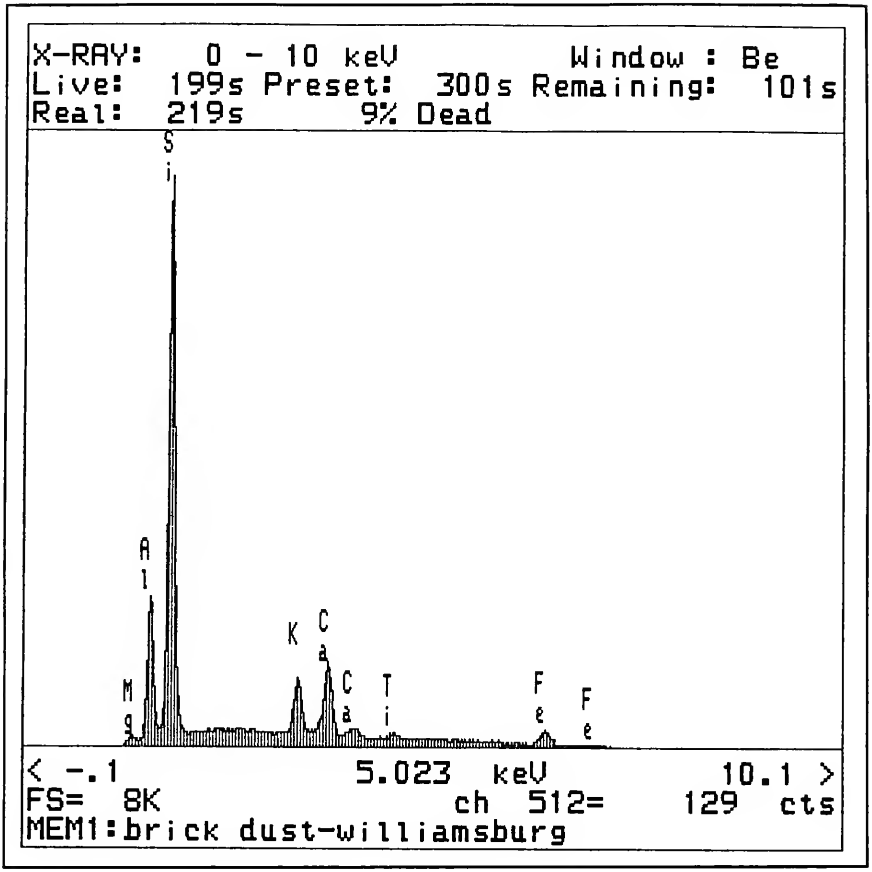


Photo 14 - SEM microphotograph, Mag x100. Brick Dust 2, (BD 2)

Particles are subangular to subrounded surface. They range in size from 300 µm to 75 µm (based on sieve analysis).



Graph - Elemental Spectragram, Brick Dust 2, (BD 2)

Elemental analysis of brick dust 2 indicates silicon as the primary constituent. Other accessory elements detected include aluminum, calcium, iron, maganese and titanium. BD 2 is similar in general composition to BD 1.

Mix 1

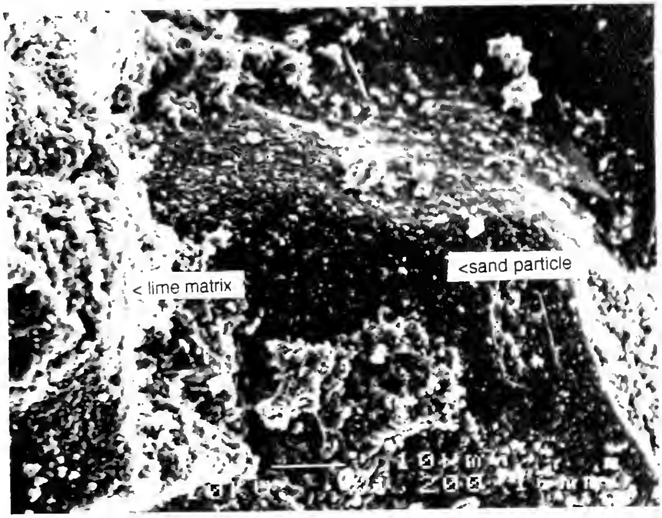
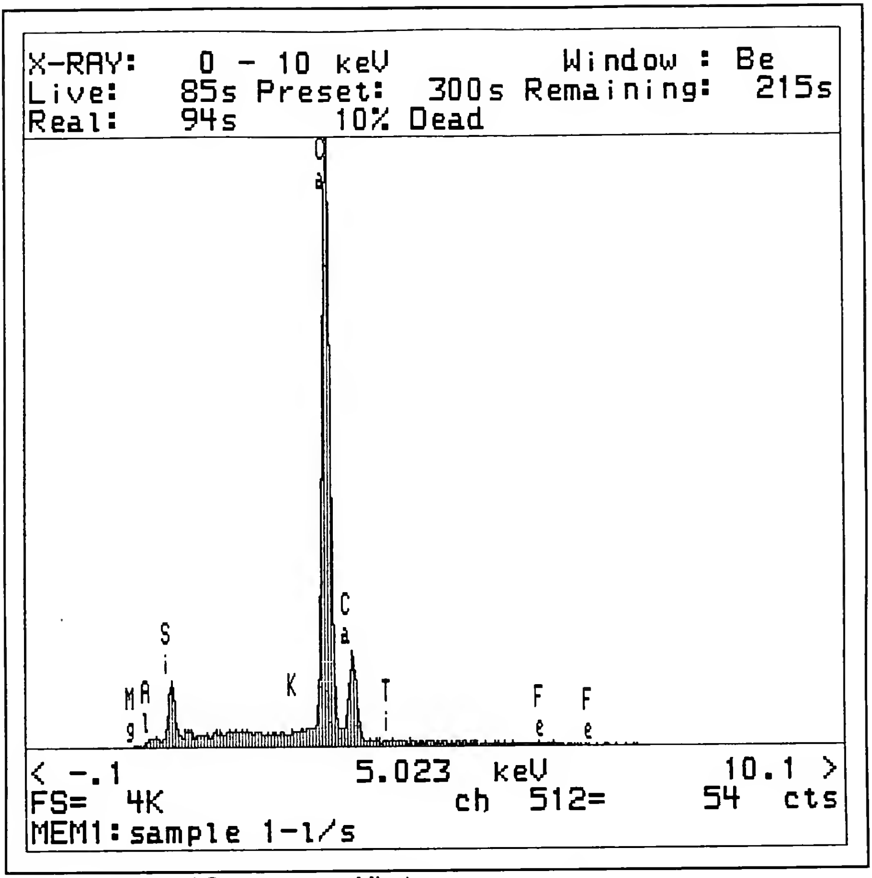


Photo 15 - SEM microphotograph, Mag. x100, Mix 1 (1 part lime to 3 parts sand)

Interface between sand particle and the lime paste matrix.



Graph - Elemental Spectragram, Mix 1

Elemental mapping of the lime and sand mix reveals a high content of calcium attributed to the lime paste binder. Trace quantities of magnesium, aluminium, silicon, potassium, titanium, and iron can be attributed to impurites in the lime and the sand.

Mix 2

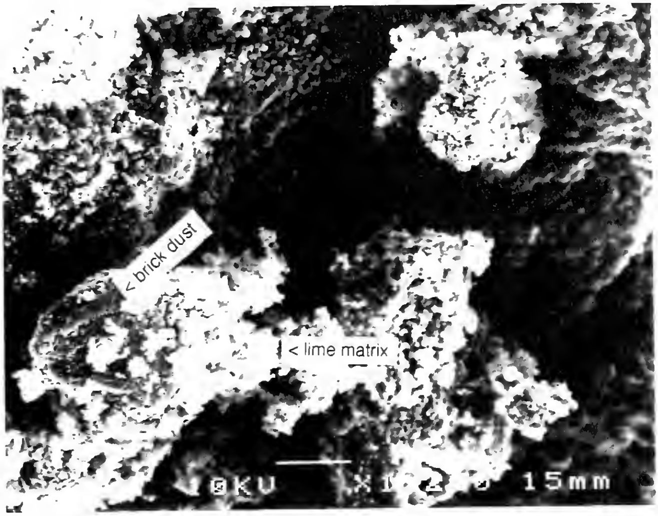
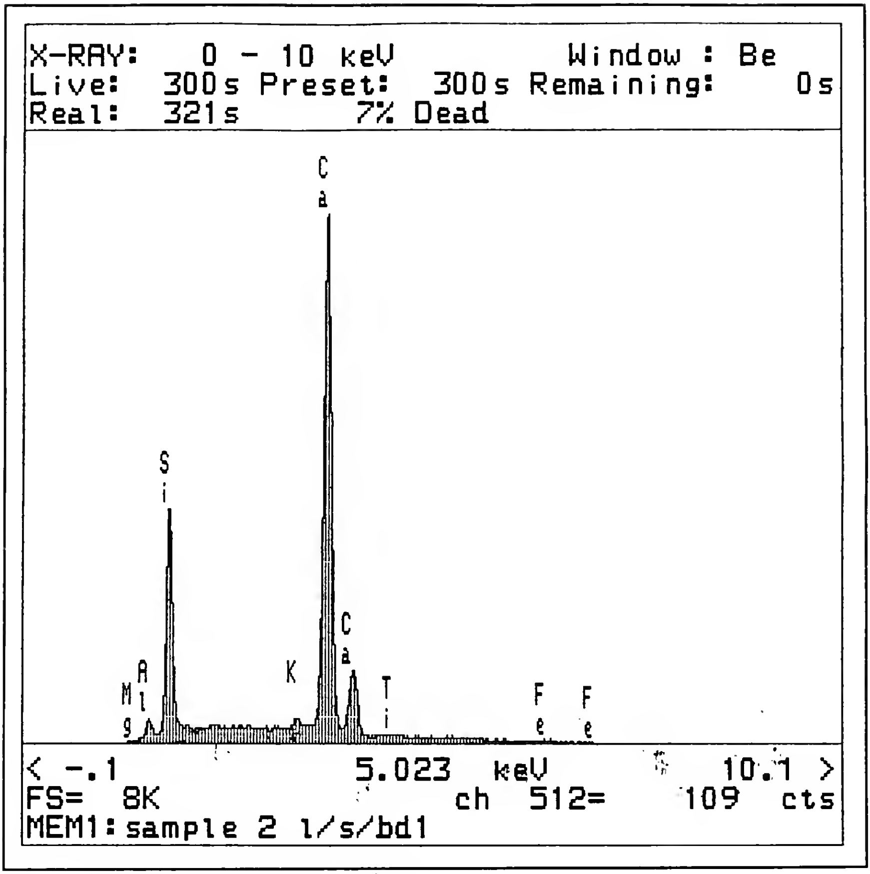


Photo 16 - SEM microphotograph, Mag x1200, Mix 2 (1 part lime, 3 parts sand and 1 part brick dust)

The addition of brick dust to the lime mixture appears to result in a microstructure different than mix 1. The particles of brick dust appear to be covered with lime, which bridges the interstitial space between the sand particles.



Graph - Elemental Spectragram, Mix 2

The addition of brick dust to the lime and sand mix is demonstrated by the large silicon peak and an increase in the presence of aluminum.

Mix 4

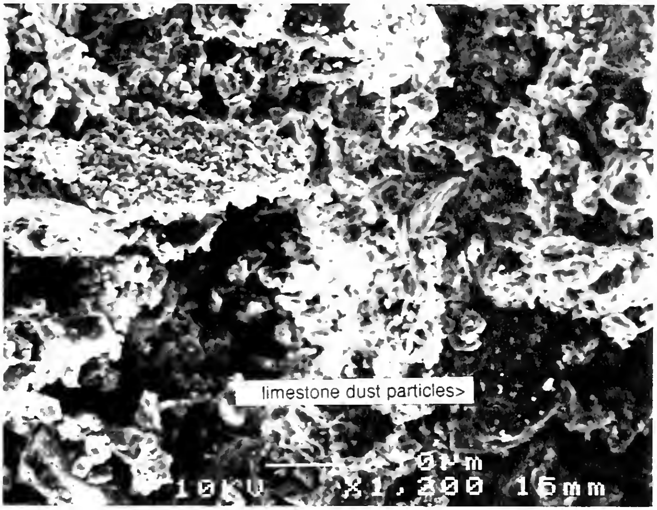
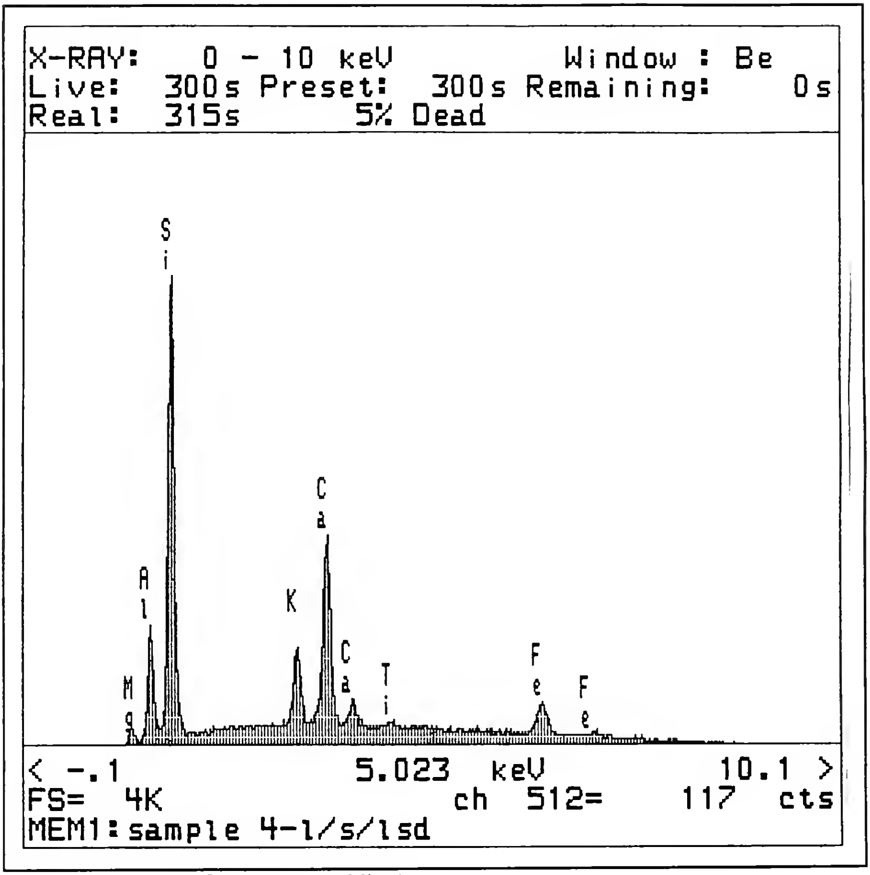


Photo 17 - SEM microphotograph, Mag x1200, Mix 4 (1 part lime, 2.5 parts sand and 1 part limestone dust)

Here limestone particles are clearly discernable and do not appear to have good contact with the lime binder..



Graph - Elemental Spectragram Mix 4

Elements detected include silicon, calcium, aluminum and potassium. The silicon is contributed from the sand in the mix.

Mix 5

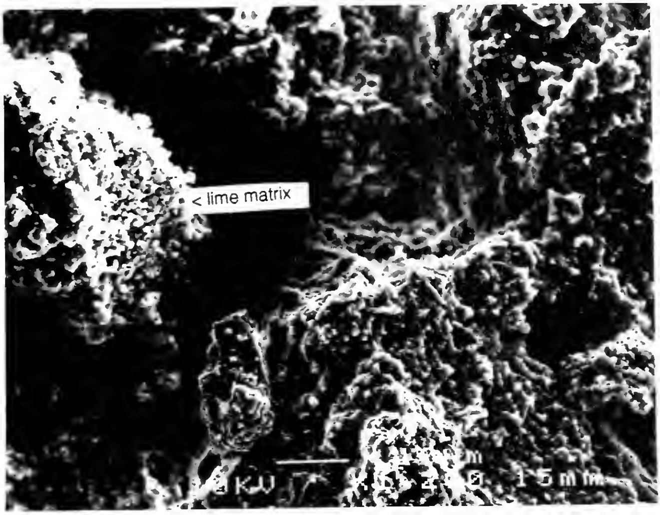
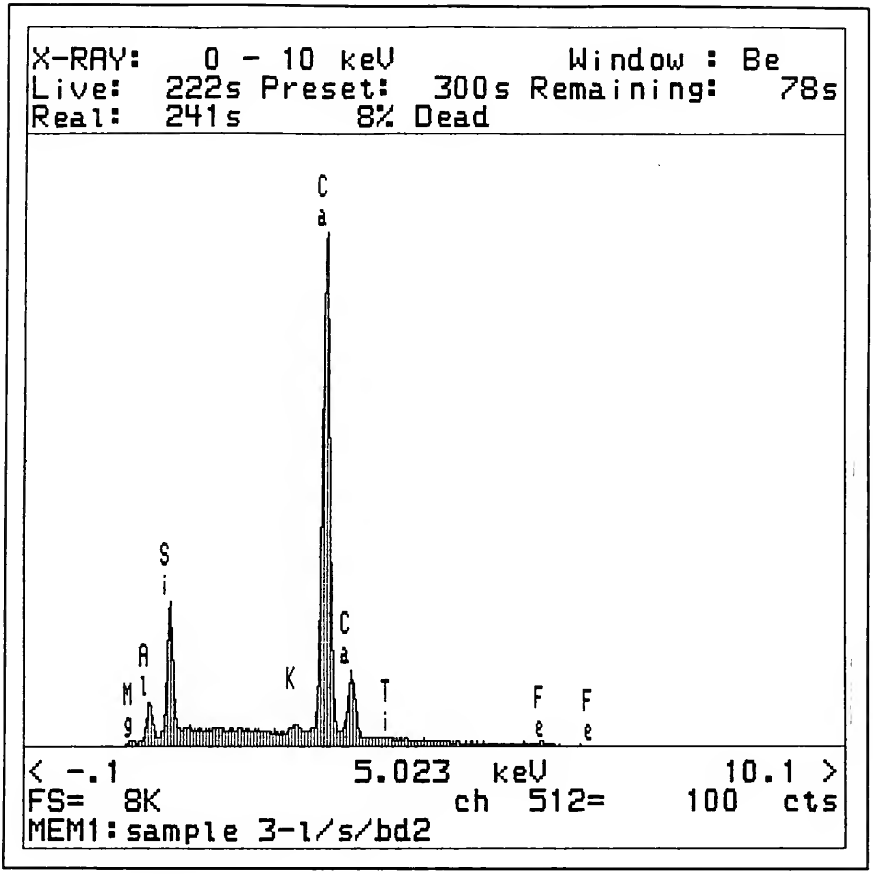


Photo 18 - SEM microphotograph, Mag. x1200, Mix 5 (1 part lime, 2.5 parts and 1 part brick dust 2)

Brick dust particles can be clearly identified in the lime matrix. As demonstrated in mix 2, the brick dust particles are covered by the lime. The lime appears to be strongly attracted to the brick dust.



Graph - Elemental Spectragram, Mix 5.

The elements identified in this mix reflect those in Mix 2. Peaks representing calcium and silicon dominate, while aluminum is also detected.

2.6.15 Porosity as Measured by Pore Size Distrubution

Studies have shown that the porosity of cured mortar is an important property as high porosity leads to poor strength and adhesion and low porosity leads to poor frost or salt resistance. The right level of porosity has been established at approximately 15% ± 3% in order to achieve high frost resistance, good workability and bond strength.[131]

The porosity of a mortar is a function of binder type, aggregate, mixing, water content and curing conditions. As an attempt was made to keep these factors consistent amongst the mixes, any differences in porosity should be a result of the addition of the brick and the limestone dusts.

The porosity of a mortar can be determined by the use of a mercury porosimeter or can be measured optically using microscopy. For the purposes of this experiment, porosity was determined by measuring the pore size distribution of a given area of the mortar sample.

Measuring the pore size of a sample differs from measuring microcracking, in that the cracks are long and narrow whereas voids are open space of any shape and size. The measurement is expressed as a percentage of a given area of the sample. A certain amount of operator error does exist, but more than one sample area was observed and the average measurements were calculated.

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[131] W. H. Harrison and G. K. Bowler, "Aspects of Mortar Durability, " Transactions and Journal of the Institute of Ceramics, August 1989, 6.

Methodology - After a 120 day period of curing, thin sections of the samples were made. Thin sections were observed under plane polarized transmitted light and the pore sizes were measured using a grid micrometer. Pore size was measured using Martin's diameter defined as the dimension that divides a randomly oriented pore into two equal projected areas. Martin's diameter is the simplest means of measuring and expressing the diameters of irregular pores and is considered sufficiently accurate when averaged for a large number of pores. Using a calibrated grid micrometer, the pore size was measured. This procedure was repeated in three representative areas for each sample and averages were calculated and expressed as percentages of a range of pore sizes found in the total area studied.

Apparatus - 1) prepared thin section, 2) polarized light microscope, 3) grid micrometer.

Results

Table 19 - Mean Pore Size Distribution of Mortar Mixes - Expressed as a %

Mix	1- 5 μm	6 - 9 μm	10 -14 μm	15 -20 μm
Mix 1 (lime & sand)	35%	42%	14%	7%
Mix 2 (lime, sand & BD1)	46%	30%	23%	0
Mix 3 (lime, sand & BD1)	39%	35%	19%	7%
Mix 4 (lime, sand & LSD)	21%	22%	27%	30%
Mix 5 (lime, snad & BD2)	64%	24%	11%	0

Table 20 - Mean % Porosity as measured by Pore Sizes of Mortar Mixes

Mix	%Porosity
Mix 1 (lime & sand)	17%
Mix 2 (lime, sand & BD1)	14%
Mix 3 (lime, sand & BD1)	16%
Mix 4 (lime, sand & LSD)	20%
Mix 5 (lime, sand & BD2)	14%

Discussion of Results

Although a certain margin of error exists by calculated the pore size and pore size distribution optically, error was slightly reduced by examining three different areas of the sample and expressing the results as an average. An examination of the results of pore size distribution, Table 19, reveals that the addition of brick dust to the mixes does reduce the size of the pores. As more fine particles are added to the mixture the spaces and sizes of the spaces are reduced. In the case of Mix 4, larger pore size were found even though the more fine particles were added to the mix. The pore sizes of Mix 4 are probably related to the high rate of microcracks exprerienced in this mixture.

In calculating the percent porosity based on pore sizes, the mixes tend to fall within the acceptable level of 15% ± 3%. Mix 4 falls just outside of this level, however, it has been established that this mix was hampered from a low water content during initial mixing.

2.7 Conclusions

In this experimental program certain trends in the behavior of brick dust added to lime-based mortars have been witnessed. Although the results do not indicate significant changes in behavior of the lime-based mortars, statements can be made about the role of brick dust as a pozzolanic additive. As pozzolanas must be tested in combination with other materials, many variables exist that hinder the formulation of tangible conclusions.

A certain degree of uniformity was achieved in the mixing, curing, and standardized testing which reduced many of the variables in this research. However, a margin of error existed as many of the tests were subjective or involved using non precision equipment. To reduce this margin of error statistical analysis was conducted when possible.

The results of the experimental program appear to be the first quantifiable data produced on the properties of these North American mortar materials. Future testing on these or related materials can use these results for comparative purposes. The results have potential implications for related research, for example future phases of the Smeaton Project. The materials, the selected standardized testing and the results could serve as a guide for future avenues of research. Similarly, the problems incurred in this experimental program could be avoided.

In terms of the materials selected for the program, it can be stated that the lime putty and both types of brick dust demonstrate promise for use in the repair and conservation of historic structures. Although the focus of this

research was not to evaluate the lime putty, Mix 1 did perform well in its resistance to salt attack. Both types of brick dust appear to impart pozzolanic properties as witnessed by experiments like setting rate and depth of carbonation and observations like microcracking and pore size distribution. BD 2 appeared to perform slightly better than B D 1, on many of the comparative tests such as setting rate, water vapor transmission, depth of carbonation. As well BD 2 appeared to exhibit less microcracking than did BD 1. At this time, this difference can not be explained.

The differences in proportioning between Mix 2 (1:3:1 - lime:sand:BD) and Mix 3 (1:2 1/2:1 - lime:sand:BD), did not appear to impact their behavior in the experimental program. At the onset of the research, it was thought that the tests and examinations would be discreet enough to observe behavioral differences in the mixes. The selected program did not detect any distinguishable trends due to the difference in proportioning as the difference was too small.

Mix 4, modified with limestone dust rather brick dust, was included in the experimental program to evaluate whether the brick dusts were behaving as a pozzolana and/or a porous particulate. As porous particulate may have ameliorating affects on lime-based mortars, it is important to juxtapose the behavior both materials impart on lime mortars. Clearly a distinction could be made between the properties imparted by the two different materials. In terms of setting rate, depth of carbonation, and microcracking, the brick dusts distinguished themselves as imparting pozzolanic properties. However, it was discovered at the end of the testing program that the limestone dust may have been too small to behave as a porous particulate. In the case of this

experiment it did serve to distinguish the role of the addition of the brick dust to the lime based mortar as a control.

This experimental program did serve to evaluate the available North American standardized tests for mortars. Many of these tests are intended to evaluate cement-based materials and not lime-based materials. It appears that these two materials can not be measured with the same scale, as lime-based and cement-based mortars do not have the same properties. Tests that clearly demonstrated this observation are those that measure shrinkage and flow. Both tests yielded values that could not be used to distinguish the properties of the materials. In future testing programs, these tests should be substituted by ones that can address the behavior of lime-based mortars.

The results of the experimental program were significantly influenced by the period of time available for this research. Although some comparative tests were conducted on fresh mortar, performance and durability tests were conducted after a 120 day curing period. This period of time may not have been sufficient, as pozzolanic reaction in lime-based mortars is a time dependent process. Consequently the results derived after a 120 day period may be different than those observed after a longer period of time.

2.8 Recommendations for Further Research

The completion of this experimental program has resulted in many recommendations that would improve future reseach on this subject. These recommendations include:

1) Moist cabinets should be used for the initial curing (30 days) of lime/pozzolana mortars. This procedure provides essential moisture for the pozzolanic reaction to occur and some controls over variables for better compatibility of results. As well, the experimental results will be more comparable as this procedure is practised by researchers in the field.

2) The use of the flow table as a test to indicate workability should not be used as a comparative test, but should be used to establish consistency amongst the mixes. The correct proportion of water added to the mix could be established by using the flow table, thus reducing the risk of comparing mixes that have differences in basic properties.

3) In addition to firing temperature and particle size, the mineralogy of the brick dusts should be established.

4) Some of the experiments on hardened mortar should be conducted after a longer curing period. One year of curing should result in more conclusive results.

5) Improvements need to be made to test skrinkage of lime-based materials. The present standards for cements and test methodology do not yield effective results.

6) The rate of water absorption should be compared to the rate of water evaporation, as it is important to understand how water or moisture can escape the mortar.

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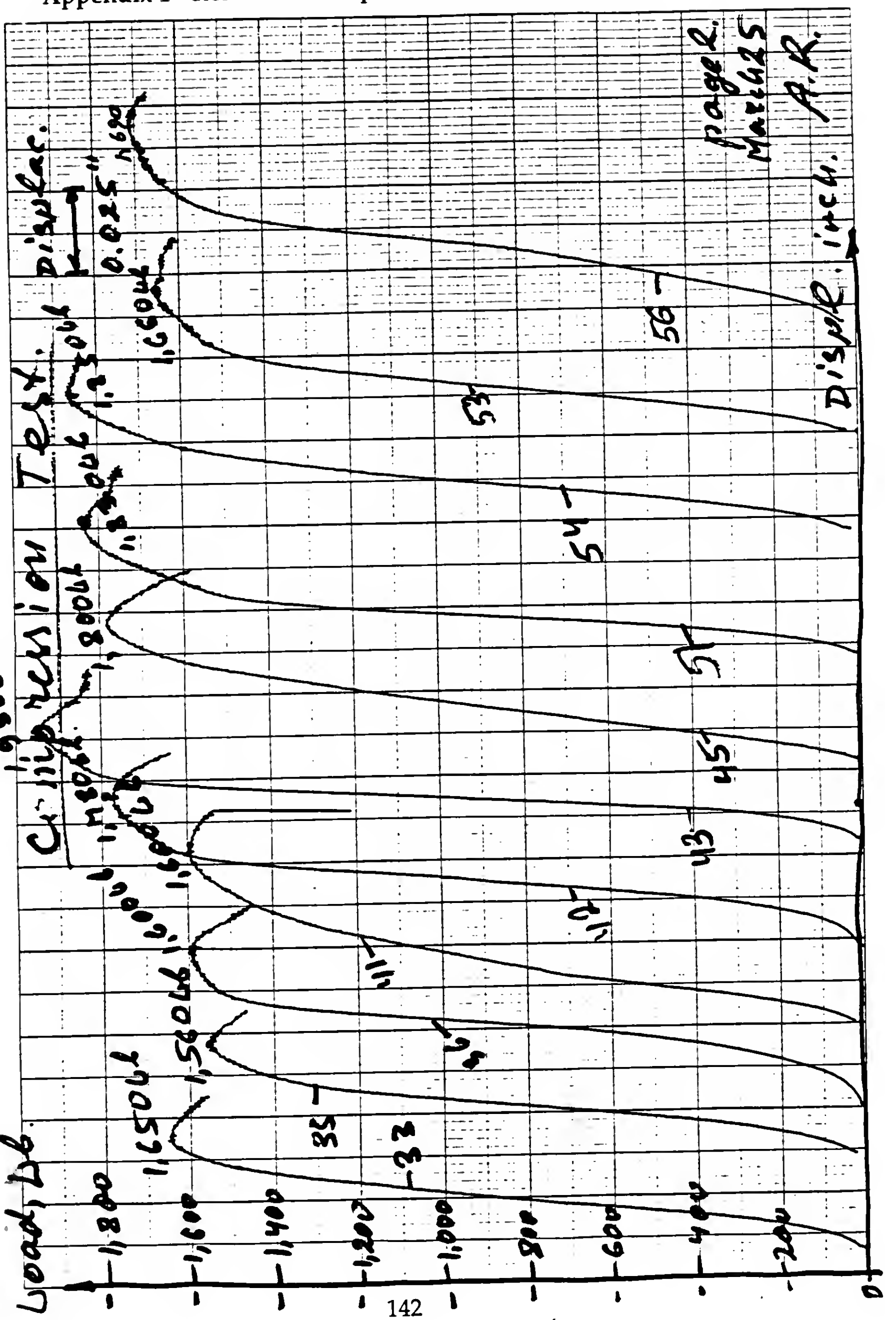
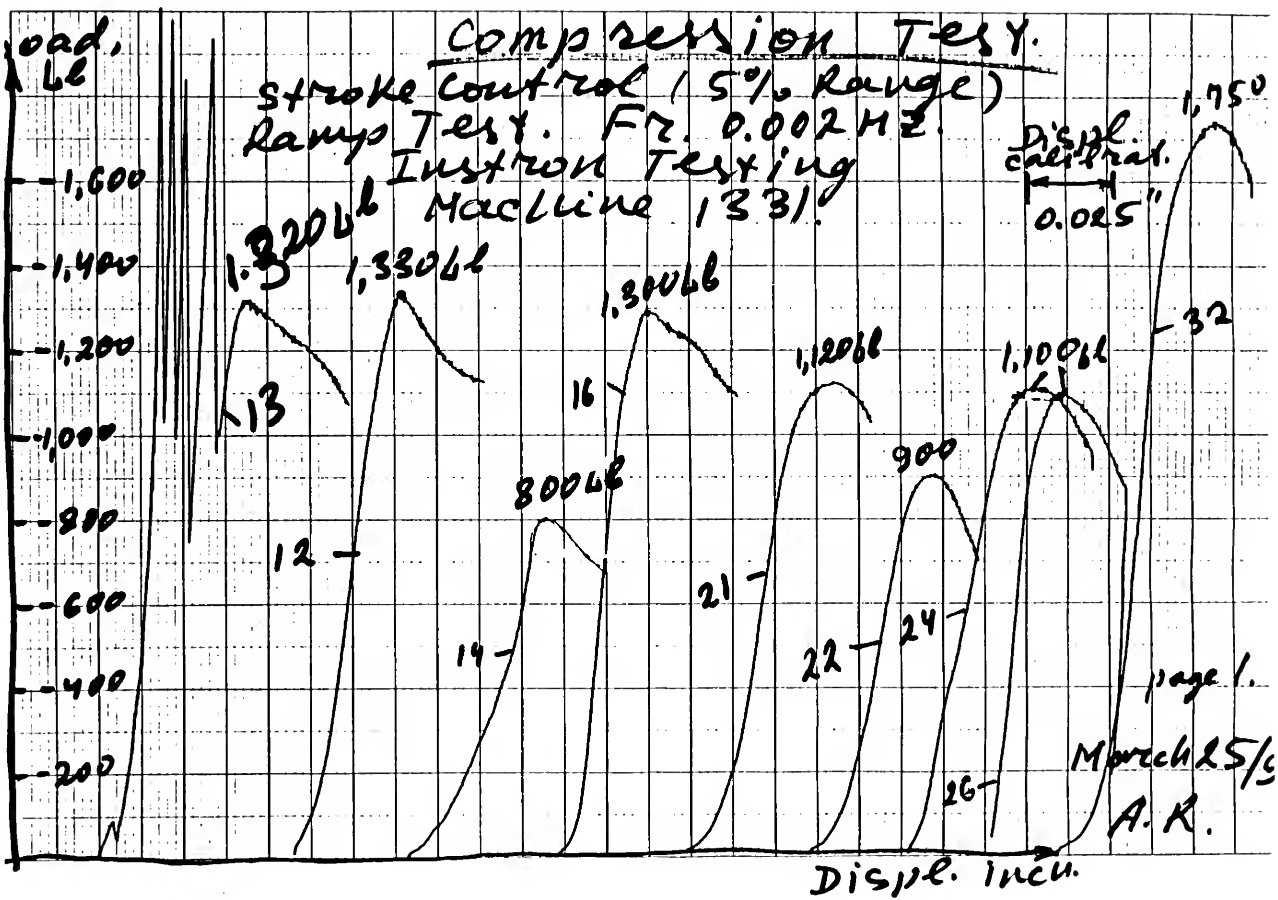
Appendix 1 - Results of Compressive Strength Testing




Compression Test.
Stroke Control (5% Range)
Ramp Test. Fr. 0.002 HZ.
Instron Testing
Machine 1331.
Load, Lb
1,600
1,400
1,200
1,000
800
600
400
200
1,320Lb
1,330Lb
1,300Lb
1,120Lb
1,100Lb
1,750
800Lb
900
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page 1.
March 25/
A.R.
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