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Evaluation and Testing of Brick Dust as a Pozzolanic Additive to Lime Mortars for Architectural Conservation

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Abstract
The purpose of this research is to identify an optimal methodology for determining whether a given brick dust will produce a pozzolanic reaction when combined with lime. This property will be referred to as *pozzolanicity*. The research required a review of the properties of pozzolanic materials, the nature of the pozzolanic reaction, and a review of existing methods for determining pozzolanicity. A testing program performed at the Architectural Conservation Laboratory at the University of Pennsylvania was designed and executed to evaluate methods for testing pozzolanicity of brick dust to determine their efficacy. An evaluation of the tests was the final result of the research, along with recommendations for ways in which this immensely valuable resource can be tested and utilized economically and sustainably for conservation work in the future.

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EVALUATION AND TESTING OF BRICK DUST AS A POZZOLANIC ADDITIVE TO LIME MORTARS FOR ARCHITECTURAL CONSERVATION

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CHAPTER 1 INTRODUCTION

1.1 LIME-BASED REPAIR MORTARS FOR ARCHITECTURAL CONSERVATION

In architectural conservation, adherence to original materials in treatments and repair is highly advantageous for the sake of material compatibility as well as authenticity and aesthetics. However, the service life of repairs is also an undeniably critical consideration for practicality and sustainability. This is particularly relevant in the formulation of repair mortars for the bedding and pointing of masonry work. A conflict exists between the ideal of using lime mortars for repair and the insufficient long-term performance associated with these materials. As a result, lime mortars have frequently been gauged with, or entirely replaced with Portland cement in the repair of historic masonry structures. Time has revealed the negative consequences of this practice through extensive damage to historic brick and stone structures, evident in the deterioration of the masonry systems because of incompatibility between soft, permeable masonry unit and mortar systems and hard, impervious Portland cement.

Lime-based mortars are optimal for conservation for several material-related reasons. Mortars are meant to be sacrificial materials that are softer and more permeable than adjacent masonry units. Lime mortars are permeable and porous, allowing the movement and evaporation of water in the liquid and vapor form, whereas Portland cement is rigid and less permeable with a very tight pore

structure. Water cannot easily evaporate through Portland cement mortar joints and is retained within the masonry unit instead. This causes weathering and mechanical damage through freeze-thaw cycling, as freezing water expands within the pores and causes stresses and the breaking up of a masonry unit over time. Mechanical damage is also inflicted on the stone as a result of crystallization of soluble salts trapped in the stone by impermeable cement through wetting and drying cycles. Portland cement can aggravate mechanical damage from salts because it introduces salts itself, particularly gypsum. Freeze-thaw and salt damage can be avoided with the use of a permeable mortar that allows evaporation through the sacrificial joints as opposed to the masonry unit itself. In addition to freeze-thaw and salt crystallization damage, the high bond strength of Portland cement mortars negatively impacts soft masonry units because it doesn't allow movement (structural or thermal) and it also causes damage to the stone when it is removed.[1] Finally, Portland cement has a lot of inherent problems that should not be introduced into an historic masonry system if it can be avoided. For example, ettrignite is a harmful compound that is formed from the hydration of Portland cement. The crystal growth of ettrignite can cause stresses and cracking in mortar joints.[2]

The recognition of the negative consequences of the use of Portland cement mortars for repair of historic structures became apparent and, to some

[1] Pat Gibbons, *Technical Advice Note*: *Preparation and Use of Lime Mortars,* Revised edition 2003, The Scottish Lime Centre. Edinburgh, Scotland: Crown Copyright, 2003, pp. 1-4.
[2] Jochen Stark and Katrin Bollmann, "Delayed Ettrignite Formation in Concrete," (paper) Bauhaus University, Germany.

extent, initiated interest in the return to traditional practices of lime mortar pointing for conservation projects in the last decades of the twentieth century. While lime mortars are ideal in terms of material compatibility, their less-than-optimal durability (resistance to extreme weathering) requires a maintenance program that is often too intensive to be economically viable for the custodians of cultural heritage. Their curing time also affects their viability, as lime mortars, particularly when deprived of carbon dioxide within a wall or between masonry units, can take many years to fully cure. Recent investigations into the properties of lime mortars have broadened the understanding of these materials and sought to make them more practical. One study that has been particularly insightful is the Smeaton Project, initiated by English Heritage in response to inappropriate Portland cement repointing of Hadrian's Wall in northern England. An important finding was the confirmation that the addition of brick dust to pure lime mortars greatly enhanced their performance because it acted as a *pozzolan*.[3]

1.2 POZZOLANS: THEIR ROLE IN LIME MORTARS

A pozzolan can be simply defined as a material that contains reactive silica and/or alumina that, when combined with lime, will react to form new compounds (calcium silicate hydrates and calcium aluminum hydrates) that have the ability to modify the properties of a lime mortar. Specifically, the addition of a

[3] Jeanne Marie Teutonico, Iain McCaig, Colin Burns, John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-based Mortars," *APT Bulletin*, Vol. 25, No. 3/4. (1993), pp. 32-49.

pozzolan enhances the properties of lime mortar by speeding setting time, increasing strength and long-term durability, and allowing a hydraulic set; the property of a mortar to cure under water without the action of carbon dioxide. Essentially, the addition of a pozzolan to pure lime is a way of artificially creating a material akin to hydraulic lime. Hydraulic lime is a material that naturally possesses the ability to set quickly in the presence of high moisture because of reactive silicates contained in the mineralogical clays of the limestone from which it is derived.

Sources of pozzolans include a diverse range of materials, some naturally-occurring and some artificial. The use of pozzolans in lime mortars can be traced back to Ancient Rome and earlier. A volcanic sand known as *pozzolana* was employed by Roman engineers in their mortars to form Roman concrete centuries before the invention of Portland cement-based concrete. The construction of some of the most monumental and iconic ancient structures, including the Collosseum and the Pantheon in Rome, were made possible by the strength and durability imparted to lime mortars by pozzolans. The longevity of these monuments is a testament to the quality of the materials employed in their construction.

While (volcanic) *pozzolana* was the primary additive to lime mortars in ancient Rome, crushed brick was used as an alternative when *pozzolana* was not available. This is cited in Vitruvius's *Ten Books on Architecture* and has been evidenced in analysis of samples of ancient mortar found throughout the Roman

Empire.[4] This building technology was not only utilized by the Romans, but is known to have been used in many different parts of the world. In India and Egypt, the practice of adding burnt clay to lime mortars goes back centuries and is known in these countries as Surkhi and Horma, respectively.[5] In the 1750s, Englishman John Smeaton famously experimented with fired clay mortars but ultimately used Italian *pozzolana* in combination with lime in the construction of the Eddystone lighthouse off the coast of Devon, England. With increased use of hydraulic limes and the eventual invention of Portland cement in the early nineteenth century, the use of pozzolans saw a rapid decline.[6]

1.3 IMPLICATIONS OF POZZOLANIC LIME MORTARS IN CONSERVATION

A renaissance in the use of pozzolanic lime mortars, particularly brick dust mortars, could be very valuable in conservation practice, not only for reasons of material compatibility and authenticity but for economy and sustainability as well. While true Italian *pozzolana* and other natural pozzolans are only available in certain regions, brick produced from fired clay is a material that has been used almost everywhere for construction throughout history in the majority of the civilized world. Through the recycling of unused brick or brick from demolished

[4] G. Binda, L. Baronio, and N. Lombardini, *The Role of Brick Pebbles and Dust in Conglomerates Based on Hydrated Lime and Crushed Brick*, Seventh North American Masonry Conference, University of Notre Dame, Indiana, 1996.
[5] F. M. Lea, *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971, p. 419.
[6] Jeanne Marie Teutonico, Iain McCaig, Colin Burns, John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-based Mortars," *APT Bulletin*, Vol. 25, No. 3/4. (1993), pp. 32-49.

structures, this abundant material can be put to a new use in lime mortars. This is a true demonstration of the concept of sustainability. Existing bricks contain embodied energy, i.e. the energy that was consumed to create each individual brick and build the structure for which they were conceived. When the bricks are no longer needed for whatever purpose they were created, their useful life can be extended by repurposing them in pozzolanic lime mortars.[7] Another aspect of the sustainability of this practice is the environmental benefit that comes from using pozzolanic lime mortars over Portland cement mortars. The process of firing Portland cement is extremely energy-consumptive and generates a huge amount of carbon dioxide. Creation of pozzolanic limes consumes significantly less energy than industrial quality limes and Portland cement.[8]

Brick dust mortars are not only sustainable, but they are also an economical option for conservation as compared to the alternatives. Natural hydraulic lime is it not economical because it is very expensive as a result of its scarcity, as it is only produced in certain parts of the world that contain sources of silica-rich limestone. In some countries, if the material is even available, the cost of purchasing and shipping it to job sites can be exorbitant. Portland cement is also scarce and expensive in some countries, in addition to being inappropriate for conservation for reasons previously mentioned. Pure lime, however, is much

[7] The idea of recycling construction waste, specifically structural and facing bricks, has been explored to some extent in a paper by Igor Pinheiro et. al: Igor S.Pinhero, Luiz C. Montenegro, and Adriana G. Gumieri. *Pozzolanic Activity of Red Recycled Bricks.* Second International Conference of Sustainable Construction Materials and Technologies, Ancona, Italy, June, 2010.
[8] Pat Gibbons, *Technical Advice Note*: *Preparation and Use of Lime Mortars,* Revised edition 2003, The Scottish Lime Centre, Edinburgh, Scotland: Crown Copyright, 2003, p.4.

less expensive and much more accessible. With the addition of brick dust, pure lime can obtain ideal properties in repair mortars using materials that are readily available and inexpensive.

1.4 RESEARCH PROBLEM

The fines of crushed brick have great potential for use as a pozzolanic additive to lime-based repair mortars. There are obstacles, however, to the successful implementation of this technology in the field. While it is firmly established that brick dust has the ability to have a pozzolanic reaction with lime, this does not apply universally to all bricks. Brick dust's ability to react with lime depends on a number of variables which will be discussed at length in this study. The impediment that exists to practically employing brick dust is the lack of standardized methods for assessing its pozzolanicity, or ability to react with lime.

While testing methodologies for assessing pozzolanicity do exist and can be found in literature as early as the 1830s, contemporary standards for pozzolanicity determination are impractical in this capacity because they are typically very complex, time-consuming, and require expensive equipment that is not available for average low-tech and small-scale conservation project, or in the field. Also, test methods for pozzolanicity determination generally are not formulated for composites of brick dust and lime but, rather, for different types of artificial and commercially-produced pozzolans used with a Portland cement binder. As is becoming increasingly evident, there is a dire need for specialty

testing standards that are written specifically for architectural conservation and traditional building materials.

There is a need for a simple method of determining a given brick's suitability as a pozzolan in lime mortars that can be performed in the field with relative ease, while yielding reasonably accurate and reliable results. Criteria for the ideal field test would include the ability to be performed with limited equipment, limited technical proficiency requirement, and would yield results in a relatively short amount of time. This is particularly important for projects or locales that may not possess the resources or technology that is required to make a thorough study to inform the use of materials.

1.5 RESEARCH GOALS

The purpose of this research is to identify an optimal methodology for determining whether a given brick dust will produce a pozzolanic reaction when combined with lime. This property will be referred to as *pozzolanicity.* The research required a review of the properties of pozzolanic materials, the nature of the pozzolanic reaction, and a review of existing methods for determining pozzolanicity. A testing program performed at the Architectural Conservation Laboratory at the University of Pennsylvania was designed and executed to evaluate methods for testing pozzolanicity of brick dust to determine their efficacy. An evaluation of the tests was the final result of the research, along with recommendations for ways in which this immensely valuable resource can be

tested and utilized economically and sustainably for conservation work in the future.

CHAPTER 2 POZZOLANICITY

2.1 INTRODUCTION

The American Society for Testing and Materials (ASTM) defines a *pozzolan* as:

> a siliceous or alumino-siliceous material that in itself possesses little or no cementitious value but that in finely divided form and in the presence of moisture will chemically react with alkali and alkaline earth hydroxides at ordinary temperatures to form or assist in forming compounds possessing cementitious properties.[9]

In this case, the ASTM defines a cementitious material as one that sets and develops strength through a chemical reaction with water in which hydrates are formed in a reaction that is capable of occurring underwater.[10] Pozzolanic additives in lime-based mortar enable them to set without the presence of atmospheric carbon dioxide and improve performance properties of strength and durability. In order to understand the nature of the pozzolanic reaction and how it intervenes in the setting of lime mortar, it is necessary to first explore the lime cycle; the sequence of chemical changes in which limestone is calcined, slaked, and set.

[9] American Society for Testing and Materials, "C 593-95 Standard Specification for Fly Ash and Other Pozzolans for Use with Lime," *Annual Book of ASTM Standards Vol .04.01*, (West Conshohocken, PA, ASTM 2000), p. 1.
[10] American Society for Testing and Materials, "C 219-01 Standard Terminology Related to Hydraulic Cement," *Annual Book of ASTM Standards Vol .04.01*, (West Conshohocken, PA, ASTM 2000), p. 2.

2.2 NON-HYDRAULIC LIME

In the United States, limestone provides the raw material for lime. Limestone can contain up to 99% pure calcium carbonate ($CaCO_3$), but many contain impurities. Much of the limestone used for lime in the United States is a magnesium, or dolomitic, limestone that contains magnesium carbonate ($MgCO_3$). Limestone with less than 5% magnesium carbonate is considered pure or high calcium lime.[11]

When limestone is burned at a temperature between 900º and 1200º C, carbon dioxide and water are driven off to form calcium oxide (CaO) or magnesium oxide (MgO), a product known as quicklime. The next step in the lime cycle is slaking of quicklime. Slaking is the process of combining quicklime with water to produce calcium hydroxide ($CaOH_2$) or magnesium hydroxide ($MgOH_2$) in an exothermic, or heat-generating reaction. Slaked lime, in the form of lime putty or hydrated lime, is the medium that is mixed with aggregate and other additives to form mortars, plasters, and grouts. Hydrated lime is a dry hydrate powder while lime putty is a wet, plastic material. Putty results from excess water during slaking. Lime putty is traditionally left to mature for a period of time before it is used in mortars.[12]

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[11] Kerstin Elert, Carlos Rodriguez-Navarro, Eduardo Sebastian Pardo, Eric Hansen, Olga Cazalla, "Lime Mortars for the Conservation of Historic Buildings," *Studies in Conservation*, Vol. 47, No. 1 (2002), pp. 62-75.

[12] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects' and Surveyors' Association, 2002, pp. 10-14.

It is the process of carbonation that induces the setting and hardening of the plastic calcium hydroxide. Exposure to air promotes the loss of water and the slow reaction of atmospheric carbon dioxide converts calcium or magnesium hydroxide into crystalline calcium or magnesium carbonate. This reaction requires the correct balance of moisture and temperature. Carbonation can occur over a very long period of time, and it will not occur if the material is not accessible to air. Without the optimal curing conditions, it can take many years for a non-hydraulic lime to develop its full strength.[13]

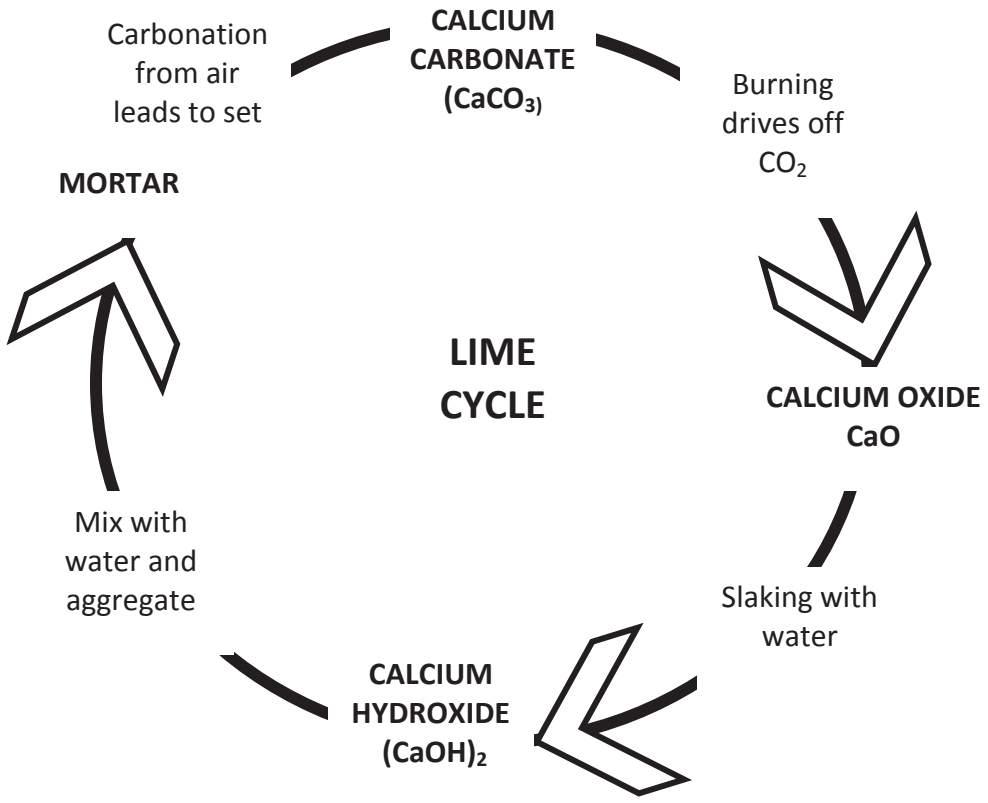

Figure 2.1 Lime Cycle (Rogers)

[13] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects' and Surveyors' Association, 2002, pp. 10-14.

2.3 HYDRAULIC LIME

The term “hydraulic” is used internationally to describe limes or cements that set through chemical reaction with water and are capable of setting under water.[14] The ASTM defines hydraulic lime as:

> the hydrated dry cementitious product obtained by calcining a limestone containing silica and alumina, or a synthetic mixture of similar composition, to a temperature short of incipient fusion so as to form sufficient free lime (CaO) to permit hydration and at the same time leaving unhydrated sufficient calcium silicates to give the dry powder, meeting the requirements herein prescribed, its hydraulic properties.[15]

Hydraulic lime differs from pure lime because it does not set entirely through carbonation but through a chemical process that allows it to set under water and without access to carbon dioxide, and it is generally faster to set and result in higher strength mortars. The raw material of hydraulic lime is different from that of non-hydraulic lime because of the presence of reactive silica, alumina, and/or clay, in addition to calcium and magnesium carbonate, in the limestone from which it is derived.[16]

When silica and clay-containing limestone is burned at a high temperature, around 1200ºC, the clay decomposes and combines with calcium to form calcium silicate ($2CaO \cdot SiO_2$) and calcium aluminate ($3CaO \cdot Al_2O_3$) and the carbon dioxide is driven off from the calcium or magnesium carbonate to leave some uncombined calcium oxide (CaO) or magnesium oxide (MgO). After

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[14] American Society for Testing and Materials, “C 219-01 Standard Terminology Related to Hydraulic Cement,” *Annual Book of ASTM Standards Vol .04.01*, (West Conshohocken, PA, ASTM 2000), p. 2.
[15] American Society for Testing and Materials, “C 141-97 Standard Specification for Hydraulic Hydrated Lime for Structural Purposes,” *Annual Book of ASTM Standards Vol .04.01*, (West Conshohocken, PA, ASTM 1997), p. 1.
[16] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects’ and Surveyors’ Association, 2002, pp. 10-14.

burning, hydraulic lime is composed of two distinct compounds: calcium silicate and calcium oxide, which will react differently when combined with water. Slaking of hydraulic lime is different from that of non-hydraulic lime because care must be taken to use the appropriate amount of water to ensure that only the calcium oxide is slaked (to form calcium hydroxide). Calcium silicates must not slake, or they will begin chemical set prematurely. If just the right amount of water is used, the free lime will slake and expand enough to break the hydraulic lime up into a fine powder that contains about 1/4 to 1/3 of its composition calcium hydroxide ($CaOH_2$) and the remainder calcium silicate ($2CaO \cdot SiO_2$). The calcium silicate may form hard clumps, known as grappiers, which must be ground and are sometimes added back into the mix to increase hydraulicity.[17]

After the hydraulic lime is mixed with water and sand to form mortars, grouts, or plasters, slaked calcium hydroxide will form hardened, crystalline calcium carbonate upon curing by carbonation in the same manner as pure lime. Hardening of hydraulic lime is achieved in part through this carbonation of free lime but primarily through the chemical reaction of calcium silicates and aluminates with water that results in the formation of calcium silicate hydrates and calcium aluminum hydrates. These cementing compounds, referred to in the cement industry simply as C-S-H, are responsible for what is known as the hydraulic set; the ability to set under water and without carbon dioxide. C-S-H

[17] Edwin Clarence Eckel, *Cements, Limes, and Plasters: Their Materials, Manufacture, and Properties*, 2nd ed, NY: John Wiley and Sons, 1922, pp. 179-180.

generically denotes a variety of calcium silicate hydrate reaction products hydrate that form fibrous networks of crystals or gels. [18]

While the term “hydraulic lime” is usually associated with lime that is naturally hydraulic, artificially hydraulic lime is another variation. During the early nineteenth century, L. J. Vicat wrote about a method of creating artificial hydraulic lime by mixing slaked pure lime with clay and calcining the mixture. Vicat called this “twice kilned,” lime referring to the process of firing the lime twice.[19]

[18] Isobel Griffin, *Grouts for the Conservation of Architectural Surfaces, Literature Review*, prepared for the Getty Conservation Institute, May 2005, pp.5-6.
[19] L. J Vicat, *Mortars and Cements,* Shaftsbury, UK: Donhead Publishing Co., 1837 (republished 1997), p. 21.

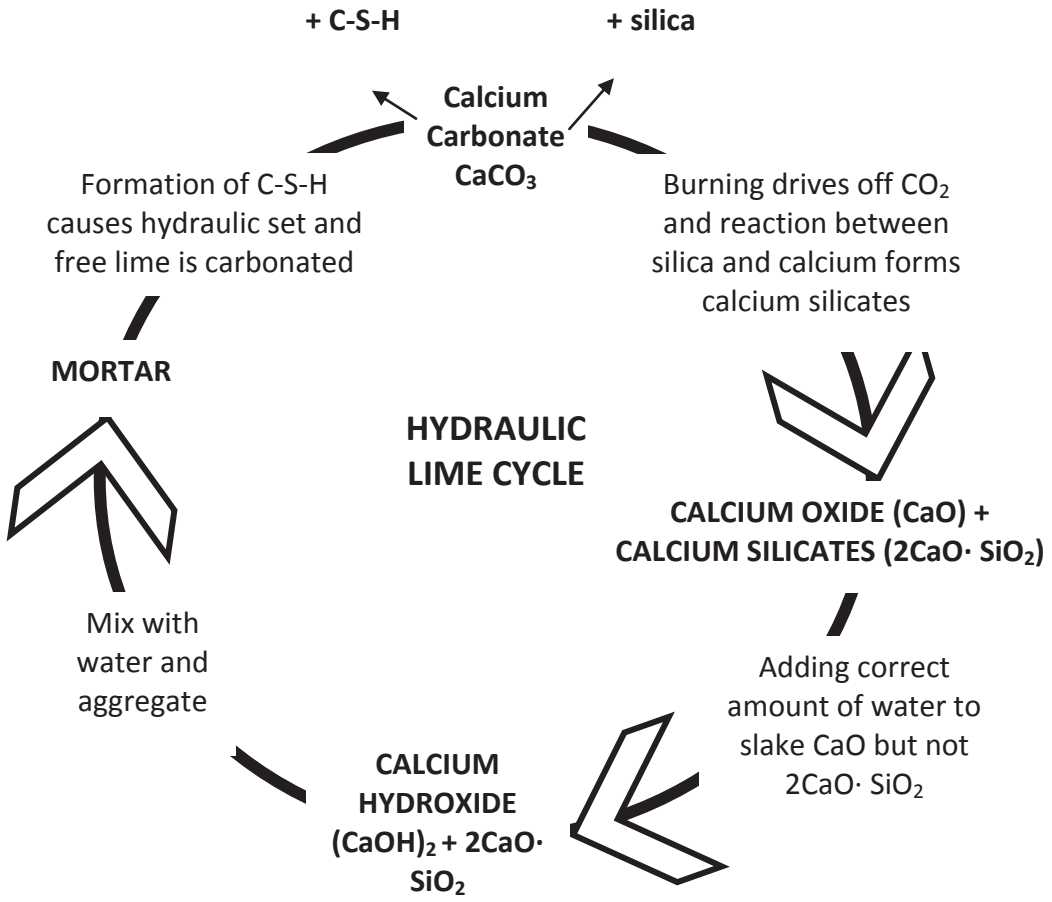

Figure 2.2 Hydraulic Lime Cycle (Rogers)

Hydraulicity is defined as the ability of a binder to harden in contact with water. The degree of hydraulicity of a hydraulic lime varies according to the chemical composition and the processing of the limestone. Generally, limes characterized as more hydraulic will cure to form harder and more impervious mortars than those of less hydraulic lime.[20] There have been several systems for classifying the hydraulicity of lime since it first began to be studied scientifically in the 19th century. Since the 19th century, the subcategories of *feebly hydraulic, moderately hydraulic, and eminently hydraulic* have been used to describe

[20] *Technical Advice Note*: *Preparation and Use of Lime Mortars.* Revised edition 2003, The Scottish Lime Centre, Edinburgh, Scotland: Crown Copyright, 2003, p. 15.

hydraulic limes. This classification system was first proposed by L. J. Vicat in 1818 and is based on clay content present in the limestone from which the lime is derived. Vicat's hydraulicity index is a ratio of the total amount of silica, alumina, and iron oxides to calcium oxide expressed as a percent. According to Vicat's theory, a higher percentage of reactive clay minerals results in a higher degree of hydraulicity. Feebly hydraulic lime has less than 12% active clay minerals. Moderately hydraulic lime has 12-18% active clay minerals. Eminently hydraulic lime contains 18-25% active clay minerals.[21]

A modern classification system developed by European Standards (BS EN 459) expresses hydraulicity based on compressive strength at 28 days. In this system, what was known as "feebly hydraulic" lime in Vicat's classification is the equivalent of NHL 2, NHL being an acronym for "natural hydraulic lime." Moderately hydraulic lime is NHL 3.5 and eminently hydraulic lime is NHL 5. Based on this classification, NHL 2 is a natural hydraulic lime that reaches a compressive strength of 2 N/mm^2 at 28 days.[22] NHL-Z is the notation for any hydraulic lime in which additional hydraulic or pozzolanic materials (up to 20% of mass) have been added. The standard also specifies the minimum amount of free lime in each classification: 3%, 9%, and 15% for NHL 2, NHL3.5, and NHL 5, respectively. NHL 2 has high elasticity, relatively low strength, and high vapor exchange. NHL 3.5 has moderate strength and is recommended when freeze-

[21] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects' and Surveyors' Association, 2002, pp. 17-20.
[22] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects' and Surveyors' Association, 2002, pp. 17-20.

thaw resistance is necessary. NHL 5 has high strength and high freeze-thaw resistance.[23]

Other less commonly used classifications exist for the characterization of natural hydraulic limes. The *cementation index* is a quantitative measurement that classifies hydraulic limes according to their chemical composition. This was developed upon the realization that Vicat's *hydraulicity index* had inherent flaws in giving the same weight to silica and alumina and, therefore, assuming that they had the same effect in producing hydraulicity. Vicat's index was also considered flawed for not taking into account the effects of iron oxides and magnesia in the hydraulic action. The cementation index is a ratio of silica, alumina, and iron oxide to lime and magnesium with a weighted value for each.[24] [25]

According to St. Astier, producers of natural hydraulic lime, the most reliable classification for hydraulicity is the theory of soluble silica. This principle relates to the amount of soluble silica available in the limestone; soluble silica is silica that can combine with calcium oxide to form calcium silicate. Silica combines with calcium oxide at a ratio of approximately 1:3 at temperatures ranging between 900º and 1000º C to form calcium silicates that are responsible for hydraulicity. A limestone containing less than 4% silica is not hydraulic, but

[23] "About Natural Hydraulic Limes," St. Astier Natural Hydraulic Lime website: http://www.limes.us/products.php, accessed April 1, 2011.
[24] Cementation index = (2.8 x % silica) + (1.1 x % alumina) + (.7 x % iron oxide)/ % lime + (1.4 x % magnesium)
[25] Edwin Clarence Eckel. *Cements, Limes, and Plasters: Their Materials, Manufacture, and Properties*, 2nd ed., NY: John Wiley and Sons, 1922, pp. 172-174.

those containing above 4% silica will be hydraulic, and hydraulicity will increase proportionately with the combined amount of silica and calcium oxide.[26]

2.4 POZZOLANS

In summary, hydraulic lime mortars develop higher strength in a shorter time period than mortars made with pure lime. They also have the property of setting under water or in locations in which they are not exposed to sufficient carbon dioxide for carbonation to occur. Pure limes that do not naturally contain reactive clay minerals may be enhanced to obtain hydraulic properties in a mortar mix through the addition of natural or artificial pozzolans. A pozzolan is a material that contains silica and/or alumina that will react with lime to form hydraulic compounds similar to those found in natural hydraulic limes. By adding pozzolans to lime mortar, the setting of the mortar will mimic that of natural hydraulic lime in that it will obtain the ability to set under water and without carbon dioxide.[27]

[26] "Hydraulicity and Properties of St. Astier Natural Hydraulic Lime," 2006, St. Astier Natural Hydraulic Lime website: http://www.stastier.co.uk/nhl/info/pdfs/Hydraulicity_and_Properties_of_NHL.pdf, accessed April 1, 2011.

[27] Geoffrey Boffey and Elizabeth Hirst, "The Use of Pozzolans in Lime Mortar," *Journal of Architectural Conservation*, Vol. 5 No. 3. 1999, pp. 34-36.

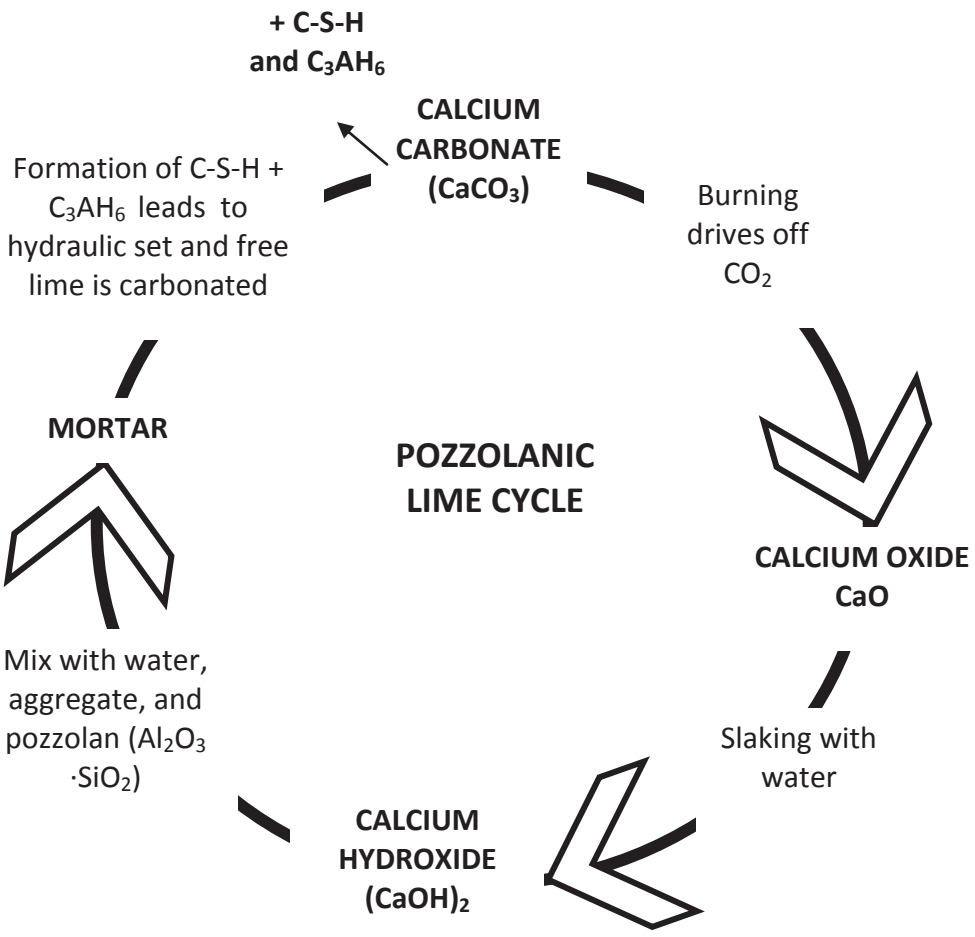

Figure 2.3 Pozzolanic Lime Cycle (Rogers)

2.4.1 USES

Pozzolans are commonly added to pure non-hydraulic lime to enhance its performance properties. Pozzolans that are finely ground to 75 microns or less have been shown to improve the properties of lime mortar by decreasing set time, allowing a hydraulic set, and increasing strength. When pozzolans are coarse (greater than 300 microns), they tend to act as a porous particulate more

than having a significant hydraulic reaction.[28] Pozzolans are also used with natural hydraulic limes to supplement strength and setting by combining with the free lime. Pozzolans exhibit a more prominent reaction when added to feebly hydraulic limes than moderately or eminently hydraulic limes. The European standard for natural hydraulic lime requires the designation “NHL-Z” for natural hydraulic limes enhanced with pozzolanic additives. Pozzolans are also used in the Portland cement concrete to increase long-term strength and combat aggregate-alkali reactions.[29]

Because pozzolanic and hydraulic lime mixes have similar properties that result from the reaction between silica and alumina with lime, pure lime enhanced with pozzolan is often referred to as hydraulic lime. Pozzolans are sometimes called “latent hydraulic” because they are not hydraulic in themselves but impart hydraulic properties and, in effect, artificially convert non-hydraulic lime to hydraulic lime.[30] According to Isobel Griffin, however, it is incorrect to use the terms “pozzolanic” and “hydraulic” interchangeably, as hydraulic materials have the ability to react with water only while pozzolans require both water and calcium hydroxide.[31]

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[28] G. Ashall, R. N. Butlin, J. M. Teutonico, and W. Martin, *Development of Lime Mortar Formulations for Use in Historic Buildings* (Smeaton Project), Proceedings of the Seventh International Conference on Durability of Building Materials and Components, 1996, p. 353.
[29] Geoffrey Boffey and Elizabeth Hirst. “The Use of Pozzolans in Lime Mortar.” *Journal of Architectural Conservation*, Vol. 5 No. 3. 1999, pp. 34-36.
[30] Paul Livezy. “Hydraulicity*.” The Building Conservation Directory. Online at http://www.buildingconservation.com/articles/hydraulicity/hydraulicity.htm.* 2003.
[31] Isobel Griffin. “Pozzolanas as Additives for Grouts: An Investigation of their Working Properties and Performance Characteristics”. *Studies in Conservation* Vol. 49 No. 1, 2004, p. 24.

2.4.2 TYPES OF POZZOLANS

There are a number of different silica-containing materials that can be classified as pozzolans. Pozzolans are divided into two classes: natural and artificial. Natural pozzolans are those that occur in nature and may or may not require calcination to become active as pozzolans.[32] Artificial pozzolans are those that are manufactured and/or must be calcined in order to become pozzolanic. Pozzolans can be further divided into the following categories based on their origin and composition.

1. Naturally-occurring volcanic ash was the first known pozzolan discovered in Italy, named for the site of Puzzuoli were they were discovered. Sometimes referred to as “true pozzolana,” they are highly reactive. These materials, which are ejected from volcanoes, have high silica and alumina contents and occur in a vitreous, finely-divided form.
2. Some types of clayey soils and crushed rock with appropriate mineral content to bring about a mild pozzolanic reaction.
3. Calcined clay products such as ceramic bricks or tiles that have been crushed into a fine powder are known to have a pozzolanic reaction with lime because of the presence of aluminates and silicates in clay. These products must be fired at a low temperature and finely ground to have a large surface area in order to be reactive. Because modern bricks are usually fired at high temperature, they normally are not pozzolanic.

[32] Geoffrey Boffey and Elizabeth Hirst. The Use of Pozzolans in Lime Mortar. *Journal of Architectural Conservation*, Vol. 5 No. 3. 1999, p. 36.

4. Fired clay products are sometimes manufactured specifically for use as pozzolans in mortars and cements. These contain highly reactive alumino-silicates that readily combine with calcium hydroxide. Metakaolin and high temperature insulation (HTI) are examples.
5. Vitreous mineral slag formed as a by-product of processes such as smelting can act as a pozzolan. Furnace slag, for example, contains reactive silica, alumina, lime, and other minerals that will produce a pozzolanic reaction with lime.
6. Organic ash materials of industrial nature are known pozzolans as well. Pulverized fuel ash (PFA) from the combustion chambers of power stations and rice husk ash are artificial and natural examples, respectively.[33]

2.4.3 CHEMISTRY OF POZZOLANS

The two primary characteristics of a pozzolan are its ability to react with lime and its ability to form reaction products with binding properties upon combing with lime.[34] Silica and alumina are the reactive components that are responsible for the combination with calcium hydroxide and formation of

[33] Pat Gibbons, "Pozzolans for Lime Mortars," *The Building Conservation Directory*, 1997, online at www.buildingconservation.com.
[34] Guilia Baronio and Luiga Binda, "Study of the Pozzolanicity of Some Bricks and Clays," *Construction and Building Materials,* Vol. 11 No. 1, 1997, p. 41.

cementitous compounds, specifically the calcium silicate hydrates (C-S-H) and calcium aluminum hydrates.[35]

The chemistry of pozzolans has long been somewhat elusive. There are a number of reaction products that can be formed as a result of the diversity of pozzolanic materials. Not all siliceous materials are pozzolanic, and there is not a clearly-defined limit for which siliceous materials will and which will not produce a pozzolanic reaction. As with hydraulic lime, the amount of silica that is soluble, or combinable, is important in predicting the formation of C-S-H. Materials with a high percentage of silica that is amorphous tend to be more pozzolanic because amorphous silica is more soluble than crystalline silica. Crystalline silica is slower or does not have a pozzolanic reaction at all and, as a general rule, the larger the crystals the less rapid the reaction. Also, calcium hydroxide and silica combine at different rates for different materials and can the reaction can sometimes be very slow.[36]

The difficulty of identifying the reaction products is exacerbated by the abundance of secondary compounds that are formed in addition to hydraulic reaction products. Chemical reactions involving alumina, iron, and alkali can result in complex compounds.[37] In 1930, G. Malquori identified $3CaO \cdot Al_2O_3 \cdot$

[35] Igor S. Pinheiro, Luiz C. Montenegro, and Adriana G. Gumieri, *Pozzolanic Activity of Red Recycled Bricks,* Second International Conference of Sustainable Construction Materials and Technologies, Ancona, Italy, June, 2010.
[36]Raymond E. Davis, *A Review of Pozzolanic Materials and their Use in Concrete,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, pp. 3-4.
[37] Raymond E. Davis, *A Review of Pozzolanic Materials and their Use in Concrete,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, pp. 3-4.

$6H_2O$ as a reaction product of the combination of lime and burnt clay. A cement symposium in 1938 asserted that a hydrated silicate with the composition of $3CaO \cdot 2SiO_2.aq$ was the reaction product in lime-pozzolan pastes. In 1940, Strätling identified a previously unknown hydrated calcium alumino-silicate that was formed by the combination of burnt kaolin and lime-water: $2CaO\ Al_2O_3\ SiO_2$. aq. (gehlenite). Strätling concluded that the reaction between burnt kaolin and calcium hydroxide was:

$$2(Al_2O_3.2SiO_2) + 7Ca(OH)_2 \rightarrow 3CaO.2SiO_2.aq + 2(2CaO.Al_2O_3.SiO_2.aq)$$

The gehlenite compound has failed to be consistently identified in lime-pozzolan mixes apart from burnt kaolinite. Another reaction product of burnt kaolin and lime was discovered by Turriziani and Schippa: $4CaO \cdot Al_2O_3 \cdot aq$ .Other pozzolans, such as trass and true pozzolana, form a hydrated calcium silicate similar to C-S-H (I) and tetracalcium aluminate hydrate when combined with lime.[38] The simplified equation for the reaction of the reactive silica (SiO_2) and alumina (Al_2O_3) with calcium hydroxide is as follows, according to Isobel Griffin:

$$SiO_2 + 4Al_2O_3 + 5Ca(OH)_2 + (x\text{-}4)H_20 \rightarrow CaO.SiO_2.H_2O +$$
$$4CaO.Al_2O_3.xH_2O$$

[38] F. M. Lea, *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971, p. 428.

with x being an integer between 9 and 13 inclusive. This reaction results in calcium silicate hydrates and calcium aluminum hydrates, and in practice some amount of calcium carbonate from carbonation of free lime as well.[39]

2.5 BRICK DUST AS A POZZOLAN

Ground bricks and other ceramic materials have been used in mortar mixes since ancient times. Although the chemistry of the pozzolanic reaction may not have been understood before the 19th century, experience had proven that the addition of powdered bricks and tiles to mortars can impart hydraulic properties. Not all bricks, however, have pozzolanic potential. The Smeaton project and other studies have been instrumental in establishing the parameters for pozzolanicity of brick regarding firing temperature and particle size. The Smeaton project proved that brick dust with a particle size below 75 microns had a greater impact on accelerating setting time and creating a higher strength hydraulic mortar. Also, the Smeaton project determined that bricks fired below 950ºC had the most positive effect on strength and durability, but was not conclusive in whether this was related to firing temperature alone or associated with the mineralogical composition of the brick.[40] In fact, the composition of clay from which brick is manufactured is a major determinant of whether it will react with lime.

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[39] Isobel Griffin, "Pozzolanas as Additives for Grouts: An Investigation of Their Working Properties and Performance Characteristics," *Studies in Conservation*, Vol. 49 No. 1, 2004, p. 24.
[40] G. Ashall, R. N. Butlin, J. M. Tuetonico, and W. Martin. *Development of Lime Mortar Formulations for Use in Historic Buildings* (Smeaton Project), Proceedings of the Seventh International Conference on Durability of Building Materials and Components, 1996, p. 353.

2.5.1 BRICK PRODUCTION

Brick is composed of various types of clay and additives such as sand and secondary minerals. Brickmaking involves a seven step process in which raw clay is converted into structural ceramic units. The clay is first mined from open pits. It is then stored in sheds with open sides to allow air drying. It is then crushed, pugged (worked with water), and extruded or hand-molded to form the shape of the brick. The clay is finally air dried before being fired in a kiln to form a hardened structural unit.[41] The composition of the brickclay will determine the quality and characteristics of the brick, as well as its potential to act as a pozzolan. As a general rule, clays containing 20-30% alumina and 50-60 % silica and the remainder consisting of magnesia carbonate, calcium carbonate and iron oxide are considered ideal for brickmaking. Clay composition is highly variable among different sources, and composition can vary significantly even among the same beds.[42]

2.5.2 CLAY MINERALOGY

There are a variety of clay types that are commonly used in brick production, and not all will have the correct mineralogy to produce a pozzolanic reaction. Clay is a fine-grained, earthy material composed of extremely small particles of clay minerals and non-clay minerals. Clay minerals are hydrous aluminum silicates, although some contain iron and magnesium rather than

[41] H. H. Murray, *Applied Clay Mineralogy: Occurrences, Processing, and Application of Kaolins, Bentonites, Palygorskite-sepiolite, and Common Clays*, Amsterdam: Elsevier publications, 2007, p. 142.
[42] Alfred B. Searle, *Modern Brickmaking*, London: Scott, Greenwood & Son, 1911.

aluminum. Some clay minerals contain alkaline and alkaline earth elements as well. Clay minerals can be classified into six groups: kaolin, smectite, palygorskite-sepiolite, illite, chlorite, and mixed-layered clays. All have different crystalline structures and chemical compositions. Each of these groups include a number different clay minerals. Most clays are composed of a variety of minerals from different groups in addition to non-clay minerals such as quartz, feldspar, mica, calcite, dolomite, opal, and others.[43]

Table 2.1 Composition of clay minerals

Clay Mineral	**Theoretical formula**
Kaolin	$Al_4Si_4O_{10}(OH)_8$
Smectite	$(OH)_4Si_8Al_4O_{20} \cdot NH_2O$
Illite	Variable- contains potassium
Chlorite	$(OH)_4(SiAl)_8(MgFe)_6O_{20}$
Palygorskite	$(OH_2)_4(OH_2)Mg_5Si_8O_{20} \cdot 4H_20$
Sepiolite	$(OH_2)_4(OH)_4Mg_8Si_{12}O_{30} \cdot 8H_2O$

Kaolinitic clay (kaolin) is a type of clay formed from kaolinite clay minerals and is the most pure of all clays; in fact it is often referred to simply as pure clay.

[43] H. H. Murray, *Applied Clay Mineralogy, Occurrences, Processing, and Application of Kaolins, Bentonites, Palygorskite-sepiolite, and Common Clays*, Amsterdam: Elsevier publications, 2007, pp. 1-4.

It is a well-defined hydrous aluminum silicate.[44] Fired kaolin (metakaolinite) has proven to be very successful in bringing about a pozzolanic reaction with lime. Many modern bricks have a low content of kaolnite and are composed, instead, primarily of minerals such as calcite, feldspar, quartz, and sodic plagioclase with small quantities of mica and clay minerals.[45] These types of bricks are unlikely to display significant pozzolanic activity when combined with lime. The most widespread clay used for production of bricks and other ceramic materials today is known as common clay. The term "common clay" is used to describe clays, shales, soil clays, and glacial clays that are used primarily for structural clay products. Common clays are fine-grained and usually plastic when wet, and they are highly varied mineralogically. Illite is the most predominant clay mineral found in common clays, but chlorite, kaolinite, smectite, and mixed-layer clay minerals are also common. Non-clay minerals including feldspar, calcite, dolomite, goethite, and hematite may be present as well.[46] Regarding composition, the ASTM and the Brazilian standards for the specification of pozzolans both require that pozzolans contain no less than 70% by weight of silica (SiO_2), aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3). The majority of clays that are used in brick production do meet this requirement.[47]

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[44] Henry J. Cowan, *The Science and Technology of Building Materials,* NY: Van Nostrand Reihnold Company, 1988, p. 140.
[45] Guilia Baronio and Luiga Binda, "Study of the Pozzolanicity of Some Bricks and Clays," *Construction and Building Materials,* Vol. 11 No. 1, 1997, p. 45.
[46] H. H. Murray, *Applied Clay Mineralogy: Occurrences, Processing, and Application of Kaolins, Bentonites, Palygorskite-sepiolite, and Common Clays*, Amsterdam: Elsevier publications, 2007, pp. 141-143.
[47] Changling He, Bjarne Osbeack, Emil Macovicky, "Pozzolanic Reactions of Six Principle Clay Minerals: Activation, Reactivity Measures, and Technological Effects," *Cement and Concrete Research*, Vol. 25, No. 8, 1995, p. 1692.

2.5.3 POZZOLANIC REACTION OF FIRED CLAY PRODUCTS

Raw clay has no pozzolanic value in itself, but when fired during the brick-making process it obtains the potential to undergo a pozzolanic reaction when combined with lime in the presence of water. When heated to temperatures between 600-950ºC, the structure of clay minerals undergoes a transformation that allows them to combine with calcium hydroxide. This is not possible, however, in clays that are mixed with high proportions of very crystalline minerals like quartz and feldspar.[48] Upon burning, the loss of combined water in clay causes the breakdown of the crystalline structure of alumino-silicates, leaving alumina and silica in an amorphous state. It is in this disordered, amorphous state that silica and alumina have the potential to combine with (and fix) calcium hydroxide. However, if calcination temperature exceeds a certain limit (generally agreed to be about 950ºC), silica and alumina will transcend the amorphous phase, recrystallize, and form other stable compounds like mullite (A_3S_2)[49] that will not combine with lime.[50] In cement chemistry, there are several abbreviations commonly used for compounds. The common abbreviations that apply to pozzolanic reactions are found in Table 2.2.

[48] Igor S. Pinheiro, Luiz C. Montenegro, and Adriana G. Gumieri, *Pozzolanic Activity of Red Recycled Bricks*, Second International Conference of Sustainable Construction Materials and Technologies, Ancona, Italy, June, 2010.
[49] In cement chemistry, the following abbreviations are used: C=CaO; A=Al_2O_3; S=SiO_2; H=H_2O
[50] Guilia Baronio and Luiga Binda, "Study of the Pozzolanicity of Some Bricks and Clays," *Construction and Building Materials,* Vol. 11, No. 1, 1997, p. 41.

Table 2.2 Cement Chemistry Abbreviations

Abbreviation	Compound
C	Calcium oxide (CaO)
A	Aluminum oxide (Al_2O_3)
S	Silicon dioxide, silica (SiO_2)
H	Water (H_2O)

Transformation into the amorphous phase follows dehydration in which the water, usually 10-15%, of the clay is burned off. This occurs at different temperatures for different clay types. One study has shown that the optimal calcination range for kaolin is between 550-950ºC, 740-920ºC for Na-montmorillonite, 650-940ºC for illite. Some clays, such as kaolin, show a sharp, sudden increase of the amorphous phase with temperature increase followed by a sharp drop in the amorphous phase at a certain temperature in which recrystallization occurs. In other clays, the increase and decline of the amorphous phase is much less abrupt. Increases in the amorphous phase can be correlated to increases in the amounts of alkali-soluble (or combinable with lime) silica and alumina. After the range of optimal calcination temperature is surpassed, the amount of alkali-soluble silica and alumina will decrease. Most clay species see a decrease in soluble alumina before soluble silica because of

the formation (with high temperature) of alumina-rich phases, like mullite, that precede the formation of silica-rich phases.[51]

Upon combination with lime, most calcined clays will form a variety of reaction products. The composition of the clay will determine the type of reaction products formed. The type and amount of reaction products are an indicator of the intensity of the pozzolanic reaction. The dominant reaction product formed from calcined clay products and lime is calcium silicate hydrate (C-S-H) and tetracalcium aluminate hydrate (C_4AH_x) in various concentrations. Alumina and silica are both present in the reaction products, but alumina tends to be less prominent in than silica. However, gehlenite hydrate (C_2ASH_8) and hydrogarnet (C_3AH_6) are other reaction products found in clays with a higher alumina content.[52]

The reaction of metakaolinite with lime can result in several different compounds which have been clearly defined. Metakaolinite is the amorphous and highly-pozzolanic product that is formed when kaolin is heated to about 600ºC during firing of brick. The following equation represents the calcination of kaolinite:

$$Al_2Si_2O_5(OH)_4 \text{ (kaolinite)} \rightarrow Al_2Si_2O_7 \text{ (metakaolinite)} + 2H_2O$$

[51] Changling He, Bjarne Osbeack, Emil Macovicky, *"*Pozzolanic Reactions of Six Principle Clay Minerals: Activation, Reactivity Measures, and Technological Effects," *Cement and Concrete Research*, Vol. 25 No. 8, 1995, pp. 1692-1700.
[52] Changling He, Bjarne Osbeack, Emil Macovicky, *"*Pozzolanic Reactions of Six Principle Clay Minerals: Activation, Reactivity Measures, and Technological Effects," *Cement and Concrete Research*, Vol. 25 No. 8, 1995, p. 1700.

When metkaolinite is combined with lime, amorphous silica and alumina can react with calcium hydrate in the following hypothetical reactions, represented in cement chemistry shorthand:

AS_2 (metakaolinite) + 6CH (lime) + 9H (water) → C_4AH_{13} (tetracalcium aluminate hydrate) + 2 C-S-H (tobermorite)

AS_2 + 5CH + 3H → C_3AH_6 (tricalcium aluminate hydrate) + 2 C-S-H

AS_2 + 3CH + 6H → C_2ASH_8 (hydrated gehlenite)+ C-S-H[53]

The type of C-S-H in the above reaction can be more specifically classified as C-S-H (I), which consists of poorly crystallized foils or platelets with a tobermorite-like structure. The exact chemical composition of the calcium silicate hydrates formed in the pozzolanic reaction varies with the water: solid ratio of the mix and the temperature; the composition also changes over the course of the reaction.[54]

In conclusion, bricks that are fired at a temperature below 950°C and are finely ground into a powder can bring about a pozzolanic reaction when combined with lime given that they are composed of a type of clay that has a sufficient amount of soluble silica and alumina. Soluble silica and alumina react with calcium hydroxide and water to form a variety of calcium silicate hydrates, or C-S-H, that are responsible for the hydraulic properties in pozzolanic mortars. There are many variables in determining whether or not a particular material will

[53] Guilia Baronio and Luiga Binda, "Study of the Pozzolanicity of Some Bricks and Clays," *Construction and Building Materials,* Vol. 11 No. 1, 1997, p. 45.
[54] F. M. Lea. *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971, p. 179.

have the potential to have a pozzolanic reaction with lime. The following chapters explore methods of evaluating materials to determine whether and to what extent they will have a pozzolanic reaction with lime.

CHAPTER 3 LITERATURE REVIEW

3.1 INTRODUCTION

Pozzolans have been used in mortars since ancient times and appear in texts as early as the 1st century BC with Vitruvius' *Ten Books on Architecture* which recommended the use of burned, pounded, and sifted brick in mortar for a better composition.[55] Ancient sources of literature on the use of pozzolans in mortar are abundant, but it was not extensively examined for this research. Rather, more current literature on pozzolans and, specifically testing of pozzolans, was the focus of the literature review.

3.2 EARLY RESEARCH

L. J. Vicat was one of the earliest researchers to take on the issue of characterizing pozzolans, and much of his work on pozzolans is still relevant today. In 1837, Vicat's treatise on mortars and cements addressed the use and classification of pozzolans. His classification system divided pozzolans, and other additives, into the categories of "very energetic," "energetic," "feebly energetic," and "inert." These classifications were based upon the setting time when combined with lime, hardness upon set, and the pozzolan's consistency. Vicat acknowledged the difficulty of predicting a pozzolan's reaction with lime based only on its physical characteristics. Regarding chemical composition, he noted that "those who possess chemical knowledge may apply it usefully in this case;

[55] Marcus Vitruvius, *Ten Books on Architecture*, Book II, translated by Morris Hickey Morgan, New York :Dover Publications Inc, 1960, p. 45.

for without making a rigorously exact measurement of the qualities of the above-mentioned substances [pozzolans], these agents assist us in classing them in an approximate manner…"[56] He also noted pozzolans' reaction when combined with limewater and decomposition when treated with acid. Calcined clay pozzolans, Vicat noted, would form a "very energetic" pozzolan if the clay was principally composed of silica and alumina and was of a fine consistency. Prior to Vicat's work, the hydraulic properties of pozzolans had been attributed to the presence of iron. Vicat's experiments with pozzolans disproved this theory.[57]

In 1927, a special report of the Building Research Establishment (BRE) was published on lime and lime mortars. *Lime and Lime Mortars*, (Special Report No. 9) was written by A. D. Cowper and provided a state-of-the-art review of lime materials and concretes, including the use of pozzolans. Cowper acknowledged that when a pozzolan was added to lime, the lime "will show marked hydraulic properties and a development of considerable strength on setting independent of any slow and uncertain process of carbonation, seen though a fat, non-hydraulic lime had been used."[58] Cowper also introduced a simple field test for evaluating pozzolanic materials through the visual observation of calcium silicate hydrates formed by the combination of lime and pozzolan.

Extensive investigations into pozzolans were undertaken in the United States in the middle of the twentieth century when their benefits for use in

[56] L. J Vicat, *Mortars and Cements,* Shaftsbury, UK: Donhead Publishing Co., 1997 (originally published 1837), p. 54.
[57] L. J Vicat, *Mortars and Cements,* Shaftsbury, UK: Donhead Publishing Co., 1997 (originally published 1837), p. 183.
[58] A.D. Cowper, *Lime and Lime Mortars,* Shaftsbury, UK: Donhead Publishing Co., 1927, p. 47.

Portland cement concrete as a combatant of sulfate attack and expansion due to alkali-aggregate reaction were discovered. In Europe, pozzolans had already begun being used as an additive in Portland cements. A symposium on pozzolanic materials, sponsored by the ASTM, was held in 1949 and explored many aspects of Portland pozzolan cements. At this early stage of research, the difficulty of analyzing and testing pozzolans was acknowledged. Raymond Davis noted the inability of the chemical composition of a pozzolan alone to determine its reactivity, and the lack of appropriate methods of evaluating pozzolans:

> …one of the problems which has long been under discussion and which is not completely solved is the development of a satisfactory method of test which may be employed reliably to evaluate a pozzolan within a reasonably short period of time. Our inability to judge a pozzolan except by long-time performance has perhaps been one of the reasons why pozzolanic materials have not been more widely used in this country.[59]

A paper by Moran and Gilliand, included in the symposium literature, cited various approaches for evaluating the activity of pozzolanic materials including composition, solubility, strength, uncombined lime, and insoluble residue. The authors noted that the majority of testing of pozzolans had previously been based on strength values alone, but that strength contribution was only one of the qualities desired in a pozzolan and not always the most important one. The authors were of the opinion that "…a single, short-time test will not evaluate

[59] Raymond E. Davis, *A Review of Pozzolanic Materials and their Use in Concrete,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p 4.

pozzolanic activity, particularly when any one of several properties may be desired of a given material."[60]

3.3 RECENT RESEARCH

The most current research on pozzolans has addressed both the historical use of pozzolans in mortars and has studied the properties of pozzolans and their potential for use in architectural conservation.

3.3.1 HISTORICAL USE OF POZZOLANS

Because crushed low-fired clay products (bricks and tile) have been used in mortars since Roman times, there have been several studies that have analyzed ancient brick dust mortars in order to characterize them and understand their usage from an historical perspective. In 1993, R. Bugini and A. Salvatori investigated the use patterns of *cocciopesto*, Italian mortars and plasters made with hydrated lime and powdered brick. The study aimed to provide insight into the differences in composition of this material that was observed in different regions and use applications. Analysis of samples from various types of sites in different regions concluded that the typological diversity of *cocciopesto* was

[60] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity*, Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 120.

intended to fulfill specific performance requirements for given purpose and was probably also influenced by practical and economic factors.[61]

Considerable efforts have been extended by Guilia Baronio and Luigia Binda to further understand both pozzolans' use in ancient mortars and the phenomenon of pozzolanic reaction between brick dust and lime. In 1988, Baronio and Binda undertook a study to analyze the composition of *cocciopesto* and to characterize the adhesion between brick and binder in brick dust mortars. The study analyzed samples taken from the Basilica di San Lorenzo in Milan. Samples were characterized through physical tests and optical examination in order to analyze the interface between brick and binder. Examinations of thin sections revealed the presence of thin, irregular layers of material of a lighter color at the interface between brick fragments and the lime binder. Analysis with electron dispersive spectrometry revealed that this reaction layer consisted mostly of silica and calcium. Binda and Baronio called these layers "reaction layers" and attributed them to a pozzolanic reaction. The study concluded that the adhesion between brick and binder is not simply physical, but due to chemical reaction as well. These chemical reactions were deduced to bring about the formation of silicates at the brick/binder interface due to silica in brick and calcium hydroxide in the binder, resulting in the bond between lime and brick dust that is responsible for the strengthening of pozzolanic mortars[62]

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[61] R. Bugini and A. Salvatori, "Investigation of the Characteristics and Properties of Cocciopesto from the Ancient Roman Period," *Conservation of Stone and Other Materials,* Proceedings of the International RILEM/UNESCO Conference, Paris, 1993, pp. 386-393.

[62] Guilia Baronio and Luigia Binda, "Characterization of Mortars and Plasters of Ancient Monuments of Milan," *The Masonry Journal,* Vol. 7 No. 1, 1988, pp. 48-54.

Another study by Binda and Baronio analyzed the role of brick dust beyond the capacity in which it strengthened mortar through pozzolanic reactions. Specifically, it examined the role of pebble-sized crushed brick in improving physical and mechanical performance in the unusually thick mortar joints (greater than 40 mm) found in some Byzantine buildings. The study involved analysis of samples taken from Byzantine buildings as well as an experimental program in which mortars were recreated and subjected to mechanical and other tests. The study concluded that, in large masonry joints, hydraulic reaction layers between brick pebbles and binder can be detected around the perimeter of the pebble where it is in contact with the binder, but this was the extent of the pozzolanic reaction and would not have served to greatly influence the strength of mortars. This suggested that there was another role for brick particles in ancient mortars besides that of pozzolanic reaction. It was hypothesized that this role might have included influence on deformability and weight.[63]

3.3.2 TESTING AND EVALUATION OF POZZOLANS

Half a century after the ASTM symposium on pozzolans, there continued to be difficulty in analyzing pozzolans and there remained a lack of standardized testing procedures. This relates to the huge variability in the reactivity of pozzolans even among the same classes of materials, but research has

[63] Luigia Baronio and Guilia Binda, "Byzantine Concretes: The Role of Thick Mortar Joints Containing Crushed Brick," *Concrete: From Material to Structure,* Proceedings of the RILEM International Conference, Arles, France, 1996, pp. 442-460.

indicated that the most influential factors include the fineness of the pozzolan and the amount of reactive silica it contains. Boffey and Hirst, in 1999, recognized the lack of testing standards for classifying and specifying pozzolans, particularly for use in architectural conservation. They noted that existing standards, ASTM for example, were appropriate for manufacture of pozzolanic mixes for new construction purposes but not aptly suited for conservation which tends to seek solutions to specific problems. They recognized the usefulness of pozzolans in conservation, but that the inexistence of pozzolans with clearly defined properties and a predictable and repeatable pozzolanic reaction inhibited their practical use in conservation projects.[64]

3.3.3 POZZOLANICITY OF BRICK DUST

Specific investigations into the pozzolanicity of brick dust for architectural conservation mortars was undertaken by ICCROM, English Heritage, and Bournemouth University in a joint research effort known as the Smeaton Project, the first phase beginning in 1993. The broad goal of the Smeaton project was to contribute to the understanding of lime-based mortars for architectural conservation. It was specifically initiated for the purpose of finding an appropriate mortar for repair of Hadrian's Wall, which had previously been repointed with a Portland cement mortar with negative consequences. In terms of brick dust, the study sought to facilitate an understanding of the effects of firing

[64]Geoffrey Boffey and Elizabeth Hirst, "The Use of Pozzolans in Lime Mortar," *Journal of Architectural Conservation*, Vol. 5 No. 3, 1999, pp. 34-40.

temperature, particle size, and proportion of binder to brick dust. The testing program included several tests on both fresh and hardened mortar in order to characterize the different mixes. These included moisture content and stiffening rate on fresh mortar and compressive strength, water vapor permeability, depth of carbonation, porosity, and sodium sulfate crystallization resistance. The testing resulted in the following conclusions relating to brick dust:

1. The addition of brick dust affects the properties of lime-based mortars, particularly in the proportion 1:3:1 lime:sand:brick dust.
2. Low-fired brick dust has the most positive effect on strength and durability of cured mortars, particularly when brick dust is a larger portion of the mix. Firing temperatures below 950˚ C are ideal.
3. Brick dust of a lower particle size range (<75 microns) reacts with lime to speed setting time and create a higher-strength cured mortar. Brick dust of a higher particle size range (>300 microns) acts as a porous particulate air-entraining additive that aids in carbonation and improves salt crystallization resistance.[65]

The Smeaton project formed the basis for future research into pozzolanicity of crushed brick. Binda and Baronio returned to their work on pozzolanicity of bricks in 1997 in response to increased interest in brick dust mortars' use in architectural conservation. The study analyzed the pozzolanicity of old and new production bricks using a pozzolanicity test for cement developed

[65] Jeanne Marie Teutonico, Iain McCaig, Colin Burns, John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-based Mortars," *APT Bulletin*, Vol. 25, No. 3/4. (1993), pp. 32-49.

by the British Standards Institute that determines pozzolanicity by saturating pozzolan with calcium hydrate to determine if the pozzolan has the capability of fixing the calcium hydroxide through reaction with the silica inherent in the material. The old bricks were sampled from two ancient buildings in Italy and the new bricks were all produced at the same plant but with varying firing temperatures above and below 900˚ C. The results of the pozzolanicity test showed that no bricks fired above 900˚ C were pozzolanic but, also, that not all bricks fired below 900˚ C were pozzolanic. The study explored the influence of clay type on pozzolanicity by performing the same pozzolanicity test on calcined kaolinitic clay and common clay that is used for ordinary brick production. Kaolinitic clay contains a large amount of kaolin and is known to be highly reactive. The common clay used in the study, however, was found to contain a very small portion of true clay minerals and was mostly composed of other minerals. The common clay showed negative results for the pozzolanicity test and the kaolintic clay showed positive results when calcined at 650˚ C. The most important conclusions of this study were that modern bricks are seldom pozzolanic because of firing temperatures above 900˚ C and that bricks made from clay that has a low content of true clay minerals do not produce a pozzolanic reaction.[66]

Another study in 1999 further explored the principles set forth in the Smeaton project. The aim of the study was to determine what factors affected the pozzolanicity of brick dust and to what extent. Using bricks produced from a

[66] Guilia Baronio and Luigia Binda, "Study of the Pozzolanicity of Some Bricks and Clays," *Construction and Building Materials*, Vol. 11, No. 1, 1997, pp. 41-46.

single clay source and a single hydraulic lime binder, the properties of particle size, firing temperature, and curing conditions (water curing and 90 % relative humidity curing) were varied in the experimental program. The compressive strength test was used to quantify the pozzolanic reaction indirectly through strength enhancement.

The study concluded that strength enhancement from brick dust was a complex function of grading, curing conditions, and the age of the sample at the time that the compressive strength test was performed. While firing temperature was found to influence pozzolanicity, fineness and curing conditions were found to be more influential on the resulting strength and optimal firing temperature was found to be a function of the brickclay's mineralogy. Also, the study found that the dependency of strength on calcination temperatures decreased over longer curing times and that those mortars made with brick dusts fired at higher temperatures (950°C) could yield optimum performance after longer periods of curing.[67]

A study was undertaken in 2004 that used scanning electron microscopy and thermal analysis to evaluate the composition and microstructure of lime mortars containing pozzolans. A variety of pozzolans were studied including Italian pozzolana, fired clay materials, kaolin, and fly ash, all combined with lime and sand in standard proportions and cured in dry and humid conditions. Both analytical techniques were carried out on both the raw materials themselves and

[67] D.B Hughes and D.C. Sugden, "The Use of Brick Dust as a Pozzolanic Additive to Hydraulic Lime Mortars," *Historic Mortars: Characterization and Tests,* Proceedings of the International RILEM Workshop, 2000, pp. 351-367.

on mortar samples cured between one and two years. Thermal analysis results yielded similar thermal curves for all pozzolanic mortars, showing a curve corresponding to weight loss from the dehydration of the calcium silicate hydrates formed around 600º C. Scanning electron microscopy differentiated the pozzolanic materials by revealing different crystal formations whose size and structure could be correlated with the mechanical strength of the mortar. This study revealed the usefulness of tools like thermal analysis and scanning electron microscopy on cured samples. It also confirmed that humid curing conditions of lime-pozzolan mortars could be correlated with improved performance. Improved mechanical strength was observed in samples cured at high humidities and could be correlated with scanning electron microscopy images depicting the growth of calcium silicate hydrates in the microstructure as well as hydraulic reaction products detected through differential thermal analysis.[68]

[68] A. Elena Charola, Paulina Faria Rodrigues, Andrew R.McGhie and Fernando M.A.Henriques, *Pozzolanic Components in Lime Mortars: Correlating Behaviour, Composition and Microstructure*, 6th International Symposium on the Conservation of Monuments in the Mediterranean Basin, Lisbon, 2004.

CHAPTER 4 MEASURING POZZOLANICITY

4.1 INTRODUCTION

The degree to which a pozzolan reacts with lime is known as its *pozzolanicity*. The pozzolanicity of a material can vary significantly, even among the same class of materials. Pozzolanicity in general is largely dependent upon the chemical composition of the pozzolan (particularly reactive silica content), the fineness of the pozzolan, and the reactivity and purity of the lime with which it is combined. The speed of the reaction is dependent upon the amount of water and the temperature.

While the classification of hydraulicity is standardized for easy comparison through quantitative hydraulicity indices, pozzolans have no universal quantitative system for classification, partially due to the diversity of sources for pozzolanic materials. Testing of pozzolans is complicated because they have no cementitious value in themselves, but only become cementitious when activated with a binder. Some tests attempt to characterize the raw material alone to determine pozzolanicity while others require the formulation of specimens in which the pozzolan is combined with a lime or cement binder. Some tests analyze the chemical properties of the pozzolan while others focus on physical and performance.[69]

[69] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 109.

4.2 TESTS FOR RAW MATERIALS

Tests on raw materials analyze the pozzolanic material alone or when exposed to lime in solution rather than as a mortar specimen.

4.2.1 CHEMICAL ANALYSIS

Analysis of chemical composition is one method of assessing a pozzolan. Because it is known that silica and alumina are the reactive components that contribute to pozzolanicity, and that pozzolanicity tends to increase with increasing content of these two components, it is logical that the degree of pozzolanicity could be assessed by determining the relative amounts of these two materials present in a pozzolan. However, because of the widely variable nature of pozzolans, it is impossible to universally classify or rate pozzolans of different classes using this method. Standards often list proportions for the minimum amount of each component, but this is more for the purpose of uniformity in specification. There are no quantitative indices based upon chemical analysis of pozzolans as there are for hydraulicity. While it is certainly useful for characterizing and ensuring uniformity in pozzolans, determination of chemical composition alone is not a conclusive test for pozzolanicity, as it has been shown to offer no definite correlation with field and laboratory behavior. Chemical composition can be determined in the laboratory through a number of chemical

tests to determine the amount of each element or compound in question, or through more sophisticated methods such as x-ray fluorescence.[70]

4.2.2 SOLUBILITY TESTS

Another chemical method of analyzing raw materials for pozzolanicity is through solubility tests. Solubility tests measure the amount of a material soluble in some medium using gravimetric methods. There are several variations that employ different reagents and test procedures, but they all are designed primarily to determine the amount of soluble silica as a measure of pozzolanicity. The method proposed by Feret in 1933 was based on the theory that a raw pozzolan is relatively insoluble, but the reaction products formed when that pozzolan is combined with lime are much more soluble. The test recommends first determining the amount of silica, alumina, and iron oxide dissolved by cold hydrochloric acid on a raw sample of pozzolan, using loss on ignition. Next, using the same amount of pozzolan, a lime-pozzolan paste is created and cured. After it is dried, it is ground to the same fineness as the original raw pozzolan and treated with hydrochloric acid to determine the amount of silica, alumina, and iron oxide rendered soluble during the hydration process. Data from these tests typically show a progressive increase in the amount of soluble silica and alumina in the lime-pozzolan mix as the reaction takes place over time. While solubility tests can be useful for comparing materials of the same class and determining

[70] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 110.

has pozzolanic potential, they lack the ability to determine the quality of a pozzolan or its degree of pozzolanicity across categories.[71]

4.2.3 LIME COMBINATION TEST

Lime combination tests measure the ability of a raw pozzolan to combine with lime as an indication of its reactivity. The test is grounded in the theory that the more reactive the pozzolan, the more it will combine with and fix lime during the reaction between silica and alumina with calcium hydroxide. This method was first proposed by Vicat in 1837. A measured sample of the raw material is placed in contact with a saturated solution of lime water (calcium hydroxide). At various time intervals, a portion of the solution is extracted and the strength of the lime is determined by titration. If the solution is highly unsaturated at the end of the test period, it can be assumed that the lime has combined with the pozzolan. In other words, the calcium hydrate has been fixed by the silica in the pozzolan, indicating a positive pozzolanicity. Volume increase in the test tube is also noted as an indication of the reaction between lime and pozzolan.[72] The most commonly accepted modern version of this test is known as the Chapelle test and appears in a French standard, NF-P 18-513: 2009 Pozzolanic Addition for Concrete.

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[71] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 110.

[72] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, pp. 109-120.

Using the Chapelle method, free lime content is determined by sucrose extraction and titration with hydrochloric acid.[73]

A similar but simplified test for determining pozzolanicity involves placing a sample of the ground pozzolan in a test tube in contact with slaked lime and water. The test tube is periodically shaken over the course of seven days. This test determines pozzolanicity not by measuring the amount of free lime remaining after the hydraulic reaction has occurred, but by visually observing the formation of calcium silicate hydrate that results from the reaction. The formation of hydrated calcium-alumino silicate compounds, which are bulkier than the pozzolan and lime themselves, and will increase the volume of solid matter and cause a retardation in the rate of settlement after shaking the test tube. These reaction products are said to have a flocculent appearance and, as a result, this test has sometimes been referred to as the "flocculation test." The simple observation of the increase in solid matter and slowed settlement rate indicates positive pozzolanicity and is also useful for comparing relative pozzolanicity of different materials.[74] This field test was suggested in a building research report of the Department of Scientific and Industrial Research in 1927 specifically for use with burnt clay pozzolan.[75] A variation of this test calls for the lime and pozzolan

[73] E. Badogiannis, G. Kakali, and S. Tsivilis, "Metakaolin as Supplementary Cementitious Material, Optimization of Kaolin to Metakaolin Conversion," *Journal of Thermal Analysis and Calorimetry,* Vol. 81, 2005, pp. 457-462.
[74] A.D. Cowper, *Lime and Lime Mortars,* Shaftsbury, UK: Donhead Publishing Co., 1927, pp. 48-49.
[75] John Ashurst. *Mortars, Plasters and Renders in Conservation.* 2nd ed., Ecclesiastical Architects' and Surveyors' Association, 2002, p. 24.

to be boiled together and then allowed to settle. The result is obtained by measuring the volume of the suspension 24 hours after the solution is boiled.[76]

4.2.4 ELECTRIC CONDUCTIVITY TEST

Electric conductivity tests exist as a time-efficient method of monitoring pozzolanic activity. As early as 1940, Frederick Lea proposed determining pozzolanicity using electrical conductivity to measure the depletion of lime from a lime-pozzolan solution as result of pozzolan fixing calcium hydroxide.[77] More recent work by McCarter and Tran has striven to produce a pozzolanicity index based upon conductivity measurements. In these tests, raw pozzolanic material is activated by dispersing it into a solution of calcium hydroxide and monitoring the chemical reaction through electric conductivity for a given interval of time while heating. Electric conductivity decreases as ion concentration of the solution decreases due to the reaction between calcium hydroxide and pozzolan and the formation of the calcium alumino silicates. A large difference in the initial electric conductivity and the stabilized conductivity indicates high pozzolanicity, and a quick stabilization of conductivity also indicates that the material is highly reactive. The rate of change in conductivity can be used as a means of quantifying pozzolanicity. The proposed pozzolanicity index is based on the

[76] Robert Day, *Pozzolans for Use in Low Income Housing*: *A State of the Art Report Prepared for the International Development Research Centre*, Ottawa, Canada, 1990, p. 58.
[77] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 113.

difference between initial conductivity and the conductivity of the solution two minutes after mixing.[78]

4.2.5 X-RAY DIFFRACTION AND SEM

X-ray diffraction (XRD) and scanning electron microscopy (SEM) have also been used in characterizing pozzolans. These methods allow the determination of whether the silica in the pozzolan is amorphous or crystalline to predict whether it will react with lime, as well as determining chemical composition in order to estimate reactivity according to proportions of silica and alumina.[79]

4.2.6 MICROSCOPY

Another method for assessing pozzolans in raw form is the use of optical microscopy. These kinds of studies can be useful in identifying and estimating the amounts of reactive constituents in a raw pozzolan, identifying mineralogical composition, and characterizing the material based on particle size and distribution, etc. When reactive constituents are amorphous and not identifiable through x-ray diffraction, they can sometimes be identified through microscopy. For example, some clay minerals and volcanic glass yield no characteristic x-ray pattern because of their amorphous structure, but can be identified visually by observation under magnification. Unless combined with other methods, optical

[78] W. J. McCarter and D. Tran, "Monitoring Pozzolanic Activity by Direct Activation with Calcium Hydroxide*," Construction and Building Materials,* Vol. 10 No. 3, 1996, pp. 179-184.
[79] Technical Brief, *Testing Methods for Pozzolans,* Practical Action: Schumacher Center for Technology and Development, online at www.practicalaction.org.

analysis cannot conclusively determine pozzolanicity. It is a useful tool, however, for supplementing other types of analyses and characterizing pozzolans.[80] Optical microscopy may be more useful in identifying reaction products in combined lime-pozzolan specimens.

4.3 TESTS FOR POZZOLAN-LIME SPECIMENS

Tests on pozzolan-lime specimens characterize pozzolans by evaluating the properties they impart to mortar mixes as an indirect measure of their pozzolanicity. Because pozzolans are only reactive when combined with lime, it is logical that their performance should be assessed in this combined form.

4.3.1 STRENGTH TEST

The most common and accepted pozzolanicity test is the strength test in which a test specimen is created using a set ratio of pozzolan to binder and the cube is subjected to compressive and/or tensile strength tests to measure pozzolanicity based on strength enhancement. The theory is that a test specimen containing pozzolanic material will have higher strength than a test specimen that does not, and that the more reactive the pozzolan, the higher the strength it will produce and the greater the discrepancy between strength of pozzolanic

[80] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, pp. 109-120.

specimen and control specimen.[81] Strength tests appear in many specifications for pozzolans and form the basis for the ASTM's Pozzolanicity Index. The pozzolanicity index is simply a ratio of the compressive strength of a pozzolan mortar mix to that of a control made without pozzolan, expressed as a percent. Lime or Portland cement can be used as the binder for this test. The Test for Pozzolanic Materials in the Indian standard uses the Lime Reactivity test, which is simply a measure of the compressive strength of a lime: pozzolan: sand mixture cured for 8 days.[82] Strength development varies with different ratios of lime to pozzolan and also with temperature and humidity during curing. Higher temperatures and moist curing conditions have the effect of higher ultimate strength, and a long period of moist curing is essential to the development of high strength in pozzolans.[83]

4.3.2 SETTING TIME TEST

Analyzing the amount of time required for a fresh lime-pozzolan mortar sample to set is another way of indirectly measuring pozzolanicity. Because pozzolans are known to speed initial and final set in a lime mortar, set time is a logical basis on which to determine pozzolanicity. A Vicat apparatus is used to test the speed at which the mortar reaches initial and final set, measuring the penetration of a needle over a period of time as the sample cures. The rapidity of

[81] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, pp. 109-120.
[82] Technical Brief, *Testing Methods for Pozzolans,* Practical Action: Schumacher Center for Technology and Development, online at www.practicalaction.org.
[83] F. M. Lea. *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971, pp. 434-435.

the set times are a measure of pozzolanicity. According to Lea, the setting time of lime-pozzolan mixes is variable. Initial set may occur in 1-3 hours but final set is usually 10-12 hours or longer.[84]

4.3.3 UNDERWATER SET TEST

A variation to the standard set time test, proposed by the French cement chemist Feret, suggests performing the setting time test on a lime-pozzolan specimen that is submerged in water. One of the properties that pozzolans impart to lime mortars is the ability to set under water and without access to carbon dioxide. Therefore, an active pozzolan should cause a set in a lime mortar under water while a non-pozzolanic control will show no set, and this is a relatively reliable indicator of pozzolanicity. The procedure recommended by Feret involves creating a lime-pozzolan paste, storing it in a glass jar covered with a layer of saturated lime water and a film of oil in order to prevent carbonation or evaporation, and measuring penetration via Vicat needle until final set is achieved. Active pozzolans will reach initial set in less than 50 hours and final set in less than 100 hours. Poor or mildly pozzolanic materials will eventually set after 100 hours, and non-pozzolanic materials will not set under these conditions.[85]

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[84] F. M. Lea. *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971, p. 433.

[85] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 116.

4.3.4 MEASUREMENT OF UNCOMBINED LIME

Another method of analyzing pozzolanic activity in a lime-pozzolan mix is to determine the amount of calcium hydroxide that remains uncombined after lime has reacted with the pozzolan in a mortar mix. This is a means of measuring the extent to which the reactive components of the pozzolan have combined with lime to form stable cementing compounds. Free lime content can be determined using a calorimetric method in which the heat of hydration is measured in order to determine free calcium hydroxide present based on the known heat of hydration of calcium hydroxide. This method has inherent errors that require correction. Another method for measuring free lime content involves an extraction of a lime-pozzolan specimen with a half-saturated lime solution. The free lime will be dissolved while calcium silicate remains.[86]

Differential thermal analysis (DTA) can also be used to determine free lime content. Observing the thermal curves of cured pozzolan-lime specimens as they are subjected to a controlled temperature program can be very useful in characterizing cured samples. Free lime content can be estimated by measuring the area under the peak caused by the dehydration of calcium hydroxide at 500-650º C. This technique can also be used to determine the presence of hydraulic components or reaction products (C-S-H). Phase changes, such as dehydration

[86] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity.* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 117-118.

of calcium alumino-silicate hydrates, are used to identify reaction products and indicators that a pozzolanic reaction has occurred. [87]

4.3.5 MICROSCOPY

Optical methods can be used for analyzing pozzolan-lime specimens as well as raw pozzolan. Petrographic-mineralogical observation of thin sections of cured mortar specimens in polarized light can provide the opportunity to visually observe reaction products formed by the interaction between calcium hydroxide and pozzolan. Reaction products appear as a thin rim of neo-formation products along the boundary between the pozzolan particles and the binder. Scanning electron microscopy can detect the composition of reaction layers. If calcium silicate hydrates are present in reaction layers, it can be concluded that the material in question has facilitated a pozzolanic reaction.[88]

4.4 STANDARDS

There are several standards produced by different organizations around the world that assess pozzolanicity by thoroughly characterizing a material through a number of different physical and chemical tests. Standards also provide guidelines and limits for chemical content, water content, and other properties. Most of these standards are more relevant for the manufacture and

[87] F. M. Lea. *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971. pp. 430-431.
[88] G. Baronio, L. Binda, C. Tedeschi, *Microscopy Study of Byzantine Mortars: Observation of Reaction Layers Between Lima and Brick Dust*, Seventh Euroseminar on Microscopy Applied to Building Materials, Milan, 1999.

specification of pozzolanic material in new construction than for conservation work where a mortar is formulated for a specific project or building.[89] Existing standards are also typically written with a focus on pozzolan use with Portland cement rather than lime.[90]

4.4.1 ASTM

The ASTM has developed several standards addressing the use of pozzolans in concrete, but ASTM C 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime*, developed in 1995, addresses the use of pozzolans with lime. This standard was written primarily for the use of pozzolanic fly ash in lime mortars, but can also be applied to fired ceramic materials such as brick dust. The standard provides a set of requirements and tests to determine a material's suitability to act as a pozzolan in lime mortars. First, the material must meet the following physical requirements:

1. No more than 10% of the material can be water soluble.
2. The pozzolan must meet a fineness requirement. A maximum of 2% can be retained on No. 30 (600 micron) sieve and maximum of 30 % retained on No. 200 (75 micron) sieve.

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[89] Geoffrey Boffey and Elizabeth Hirst. *The Use of Pozzolans in Lime Mortar.* Journal of Architectural Conservation, Vol. 5 No. 3. 1999, pp. 36-37.

[90] ASTM C618 - 08a Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete ; ASTM C311 - 07 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete ; ASTM C446 Standard Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction

3. The pozzolan must meet a minimum strength requirement when combined in a lime mortar. The minimum compressive strength is 600 psi cured at 7 days and 54 +/- 2º C and the same strength minimum at 21 days cured at 23 +/- 2 degrees C.

ASTM C 311 *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete* provides methods of determining pozzolanicity of fly ash and natural pozzolans for use in Portland cement, but some of its methods are also applicable for other pozzolans with a lime binder. The standard requires testing of the raw material for moisture content, fineness, loss on ignition, and determination of the presence of a number of different oxides through testing with specified reagents. On lime-pozzolan specimens, it analyses drying shrinkage and soundness. It measures strength through the pozzolanic activity index, a compressive strength test that compares the strength of a control with a sample containing pozzolan. Other tests are included in the specification but they apply to Portland cement but not to lime-pozzolan mixes.[91]

4.4.2 INDIAN AND BRITISH STANDARDS

The Indian standard, *Methods of Tests for Pozzolanic Materials* (IS 1727-1967), is nearly identical to the ASTM 311 *Standard Test Methods for Sampling*

[91] American Society for Testing and Materials, "C311 - 07 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete," *Annual Book of ASTM Standards.* Vol.04-02. West Conshohocken, PA, ASTM 2007.

and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete except that it also measures initial and final setting time, transverse strength, and permeability. The British Standards Institute has no specific standard for testing pozzolans, but BS EN 196-5 *Pozzolanicity Test for Pozzolanic Cements* is a procedure that is designed for pozzolanic cements, but has successfully been used in some studies on brick dust pozzolan.[92] The test is measures pozzolanicity through a lime combination test using the following method: The pozzolan is finely ground and placed in contact with a supersaturated solution of calcium hydroxide at 40˚ C for 8 days. If the lime solution is highly unsaturated at end of period, part of calcium hydroxide has been fixed by amorphous silica and test is positive. The test determines pozzolanicity by measuring the amount of lime fixed by pozzolan.[93]

4.4.3 BRAZILIAN STANDARD

The Brazilian standard ABNT NBR 12653 specifies certain chemical and physical requirements for pozzolans of three different defined classes: natural and artificial pozzolans, fly ash, and pozzolans that do not qualify as either of the other two classes. The requirements are different for each class and include a minimum combined percentage of silica, alumina, and iron oxides as well as a maximum sulfur trioxide and moisture content. It also offers specifications for

[92] L. Binda and G. Baronio have employed this pozzolanicity test in their work on analyzing brick dust pozzolan

[93] Technical Brief, *Testing Methods for Pozzolans,* Practical Action: Schumacher Center for Technology and Development, online at www.practicalaction.org.

fineness, maximum loss on ignition, maximum water content required for mixing, and strength requirements.[94]

4.5 TEST EVALUATION

The existing tests for pozzolanicity determination are varied in their complexity, expense, and method of assessment. Some testing methods could potentially be used alone to determine whether and to what extent a material is pozzolanic while others are more a means of characterization but not conclusive in themselves for determining pozzolanicity. Some tests require a considerable amount of expertise, equipment, and expense and are too complex or cost-prohibitive for a simple determination of pozzolanicity for the specification of conservation mortars. A successful field test is one that would be relatively simple to perform, inexpensive, and would yield rapid yet reasonably accurate and repeatable results. In order to design a testing program to determine the efficacy of existing methods and their potential for field analysis, the existing methods were evaluated and rated according to multiple criteria. The methods that were discussed above were examined individually to determine their suitability for a field test. They were assessed based on the following criteria:

1. Cost: Test does not require external expertise or expensive equipment or materials that would make it expensive to perform.

[94] Igor S. Pinheiro, Luiz C. Montenegro, and Adriana G. Gumieri, *Pozzolanic Activity of Red Recycled Bricks*, Second International Conference of Sustainable Construction Materials and Technologies, June, 2010.

2. Complexity: Test requires minimal steps, and is relatively simple to perform.
3. Technical proficiency: Test can be performed without extensive training and without an outside consultant.
4. Time: Results can be obtained in a relatively short period of time (i.e. less than one month).
5. Minimal equipment requirement: The test does not require extensive equipment.
6. Controlled environment: The test does not require specified temperature or humidity levels that would require that it be performed in a laboratory or other controlled environment.

For each criterion that was met, the test received one point. Tests were also evaluated based on the property measured and whether they were performed on raw pozzolan or mortar specimens, but these two categories were given no point value.

Table 4.1 Evaluation of Testing Methods

Test Method	Measurement	Sample Type	Low Cost	Low Complexity	Low Tech Proficiency	Rapid Results	Minimal Equipment	Controlled Environment	Rating
Chemical analysis (lab method)	Chemical composition	raw material	1	0	1	1	1	0	4
Solubility test	Amount of soluble silica	raw material	1	0	1	1	1	0	4
Lime combination	Ability to combine with lime	raw material	1	1	1	1	1	1	6
Electric conductivity	Rate of change in conductivity	raw material	0	0	0	1	0	0	1
Optical methods	Physical properties	raw material	0	0	0	1	0	1	2
Strength test	Compressive or tensile strength	composite	0	1	0	1	0	0	2
Time of set test	Set time	composite	1	1	1	1	1	0	5
Underwater Set	Ability to set underwater	composite	1	1	1	1	1	1	6
Free lime content	Ability to fix lime	composite	1	0	1	1	1	0	4

While the test matrix is insightful for evaluating the tests for suitability for use in the field, it cannot be used alone to judge tests, as some of the criteria have different levels of importance that cannot be weighed in the context of this simple rating system. Also, some of the tests, although they met many of the criteria for field tests, are not as practical as others because they lack the ability to yield conclusive results when used alone. For example, while chemical analysis met four of the six criteria, it is not ideal for field testing because, while it may be useful for comparing and characterizing pozzolans of the same class, chemical composition alone cannot predict pozzolanicity or quality.[95]

4.6 SELECTION OF TESTS FOR TESTING PROGRAM

Testing methods for this study's testing program were selected based on this evaluation in addition to other considerations. It was important to select tests that could potentially stand alone as a predictor of pozzolanicity and would yield conclusive results. For the purpose of comparison, it was also important to select tests that were diverse in method of assessment; to include tests that evaluated chemical and physical properties as well as tests on both raw material and mortar specimens. The following tests were selected for the testing program:

[95] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 110.

1. Setting time test: The Time of setting test using the Vicat needle met five of six criteria for a successful field test. The only unmet criterion is “no controlled environment,” as the test requires certain temperature and humidity conditions for curing. This test measures the time required for initial and final set of a mortar specimen consisting of pozzolan combined with a binder, with the rapidity of set as an indicator of pozzolanicity. Because set time is both a practical consideration when specifying mortars and an accepted means of measuring pozzolanicity, this test is ideal in that it relates directly to a necessary performance property for lime mortar.

2. Underwater Set Test: The underwater set test met all six criteria for a successful field test. This test is very similar to setting time, but it measures an essential property of a pozzolan: its ability to bring about a hydraulic set when combined with lime. This is both an important performance characteristic and a clear indicator of pozzolanicity.

3. Lime combination test: The lime combination test met all six criteria for a successful field test. While various methods for performing the lime combination test exist, this study will employ the simple test for burnt clay pozzolan proposed by Cowper in 1927. It is quick, easy to perform, and measures the ability of raw pozzolan to fix lime and produce cementing compounds, a clear indicator of pozzolanicity. This chemical test is

practical and measures an essential property of a pozzolan: the ability to react with lime to form calcium silicate hydrates.

4. Strength test: The strength test met two of the six criteria for a successful field test. The lower rating reflects the fact that it requires outside equipment and expertise in order to perform the strength tests, as well requiring a controlled environment for curing. However, as it is the most widely-accepted and standardized method of assessing pozzolans, it is an essential component of this testing program. Also, because it offers very precisely quantifiable results, it is critical for the purpose of comparison with results of other tests. As a measure of an important performance property (strength), it is a practical and logical method of measuring pozzolanicity.

CHAPTER 5 MATERIALS AND METHODOLOGY

5.1 CURRENT RESEARCH

There have been several projects performed within the Architectural Conservation Laboratory at the University of Pennsylvania that have studied brick dust mortars and grouts, either for the purpose of formulating a mortar for a specific project or site, to replicate an historic mortar, or to investigate the effects of brick dust on the properties of lime mortars. The purpose of the current research differs from past research because, rather than to formulate a site-specific mortar or to characterize brick dust mortars, it instead aims to establish a method for determining pozzolanicity of a given brick. The tests selected for the testing program are by no means comprehensive in characterizing brick dust mortars or evaluating the effects of brick dust on a lime mortar, but they were selected based on their ability to predict pozzolanic activity in a reasonably accurate and straightforward manner.

Because of time constraints and material availability, the variables in the current testing program are limited to only the type of brick dust. Particle size and proportions of various components will remain constant while mineralogical composition and firing temperature will vary inherently with the different brick dust samples. An inert control of marble dust will be used for comparison. The tests are based upon the properties of lime mortar that pozzolans are known to effect. The efficacy of determining pozzolanic potential through the selected tests is central question of this testing program. By performing identical tests on the

three different samples, the sensitivity of the test and its ability to predict pozzolanicity will be assessed. Inversely, by comparing the results from all tests for each individual formulation, consistency of tests and their ability to yield accurate results can be assessed. In addition to evaluating the tests themselves, the research should provide insight about the effect of mineralogy and firing temperature on pozzolanicity, as these are the only variables among the brick dusts.

5.2 MATERIALS

The following materials were utilized in the testing program. Data sheets, where available, are located in Appendix J.

5.2.1 HYDRATED HIGH CALCIUM LIME

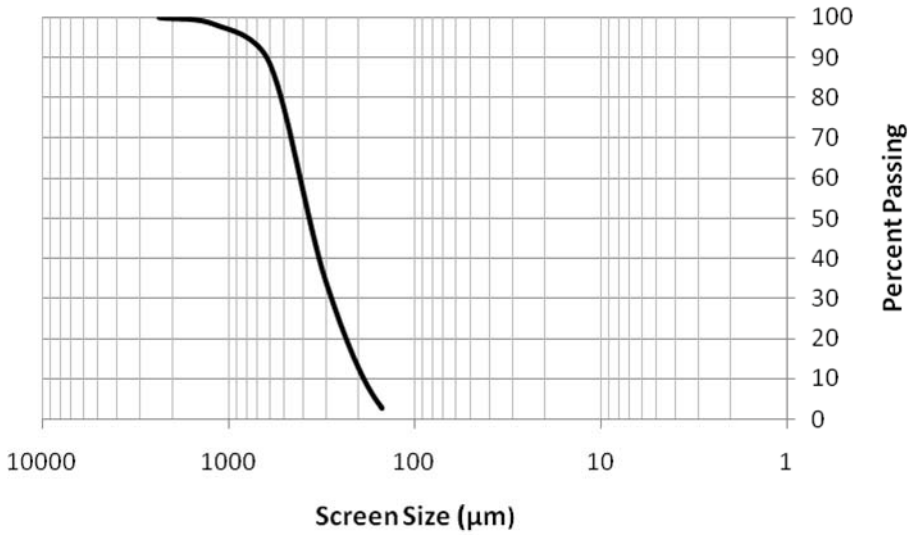
The lime selected for the experimental program is a high calcium hydrated lime donated by Coyne Chemical Company in Croydon, Pennsylvania in February, 2011 and produced by Carmeuse Lime, Inc. in Pennsylvania. The lime was produced from pure limestone containing over 98% calcium carbonate and fired in a limekiln at 900°C. During firing, the limestone was converted to quicklime. Hydrated lime was created from quicklime by adding just enough water to form calcium hydroxide. Hydrated lime is a dry, powdery product with 97% passing a 325 mesh sieve. Carmeuse hydrated high calcium lime is, on average, 76.2% calcium oxide with 24% combined water and less than 2%

magnesium oxide or silica. Although dolomitic lime is more accessible in the United States, high calcium lime was chosen as the binder for this experimental program because of its purity. The powder was stored at room temperature in a cylindrical drum. Because of the presence of clumps in the powder, the lime was sifted with a flour sifter for a more homogenous powder and for easier mixing.

5.2.2 SAND

The sand used in the mortar formulations is a bar sand purchased from CAVA Building Supply in Philadelphia in February 2011 and supplied by Dun-Rite Sand and Gravel Company in New Jersey. The sand was certified to comply with ASTM C-144 Standard Specification for Aggregate for Masonry Mortar. It is a high silica bar sand is graded predominantly between the No. 30 and No. 100 sieve. Sieve analysis was performed according to ASTM C136-01 *Standard Test Method for Sieve Analysis of Fine and Coarse Aggregates* on a 100 g sample of sand to produce a particle size distribution.

Graph 5.1 Particle Size Distribution of CAVA Bar Sand



5.2.3 MARBLE DUST

Marble dust was used as a non-pozzolanic material in order to serve as a control against which to compare the test results of the samples containing brick dust. The marble dust was purchased from Kremer Pigmente in a finely ground form with a particle size less than 32 microns. It is very pure and is composed of 95.5% calcium carbonate and about 3% magnesium carbonate and trace amounts of iron oxide, but it is void of aluminates or silicates. The purpose of the marble dust is to act as a porous particulate in the mortar formulations and as an inert control material in the other tests, because it is known not to have any chemical reaction with lime but still functions in mortar formulations as a particulate to fulfill the same mechanical function as the brick dust.

5.2.3 BRICK DUST

Two different brick dusts were used for the experimental program. Bricks were chosen based on their conformity to the parameters for pozzolanicity established in the Smeaton Project. It was essential for the selected bricks to be fired at a low temperature, within the range of 600- 950°C, as it has been proven that bricks fired above this temperature lose their ability to react with lime. While bricks were historically fired at temperatures this low, modern kilns reach much higher temperatures in order to produce harder bricks used in modern construction. It is difficult to find commercial brick producers that produce under-fired bricks. It was necessary that newly-produced bricks were used rather than recycled historic bricks in order to ascertain the approximate firing temperature. Ultimately, bricks were selected from the brickyard of Colonial Williamsburg and Belden Brick Company in Ohio.

Both sets of bricks were sent to the University of Pennsylvania in November, 2011 in whole form. They were then shipped to Puerto Rico to be crushed at San Juan National Historic Site. The bricks were crushed to varying particle sizes, ranging from large pieces 1-2 inches long to powder less than 75 microns. The crushed brick was sorted, sieved, and then the particles that did not pass the 200 (75 micron) sieve were ground with a ball mill using steel balls of various diameters until they were fine enough to pass the 200 sieve.

It was important to determine the mineralogical composition of the brick dusts in order to compare the two and attempt to correlate mineralogy and pozzolanicity, if possible. In order to identify minerals present in the dusts, x-ray

diffraction (XRD) was performed on the powdered Williamsburg and Belden brick dusts at the Laboratory for the Research on the Structure of Matter at University of Pennsylvania. A Rigaker D-Max B model with a copper sealed x-ray tube was used for analysis of samples. XRD determines mineralogy by identifying the crystalline structures of multiphase materials.

To further characterize the materials, the brick dusts were analyzed using simple laboratory tests to determine water absorption, presence and of salts (with semiquantitative commercial salt strips), acid soluble portion (through gravimetric acid digestion), pH, and color. Water absorption is expressed as amount of water absorbed by brick dust after being immersed in water as a percentage of the mass of the dry brick dust. Water absorption influences workability of the mortar mix to which the brick dust will be added as well as the water requirement. Presence of salts was determined semiquantitatively using *EM Quant* brand commercial salt test strips. Determination of salts is important because introducing salts into a mortar mix could cause mechanical damage to the mortar through the crystallization of salts as they go in and out of solution with wetting and drying cycles. pH was also determined for each brick dust using pH strips. A simple gravimetric laboratory test was used to determine acid solubility. The brick dusts were subjected to treatment with hydrochloric acid and the percentage digested by the acid was calculated. The color of the brick dusts was classified using the Munsell Soil Color Chart. The addition of brick dust to a mortar will obviously impact its color. Color is an important consideration when mortars are

used for pointing and patching in areas that will be visible, but for deep repair mortars that are not exposed, color is not usually a concern.

5.2.3.1 COLONIAL WILLIAMSBURG BRICK DUST

Colonial Williamsburg, Virginia operates a traditional brickyard in which bricks are fired in a wood kiln as they would have been historically. The Colonial Williamsburg Foundation uses these bricks for restoration and reconstruction projects on buildings within the park in an effort to utilize historically accurate building materials. They also have reported using ground brick in mortar formulations, along with lime, sand, and clay in varying proportions. Williamsburg bricks are not available for purchase commercially, but were donated for the purpose of this research. Because of their traditional method of firing, they are typical of bricks from previous centuries that might be recycled from historic buildings.

Williamsburg bricks are made from native Virginia clays. First, water is worked into the clay to form a smooth consistency. Next, the clay is cleaned and shaped into a wooden mold. The soft, unfired bricks are allowed to dry in air for about seven weeks, first on raised beds of sand then in drying sheds, before being placed in the kiln. About 20,000 bricks are stacked in the kiln. Bricks are used to build four fire tunnels. The exterior of the structure is sealed with clay and four wood fires are lit within the fire tunnels. The fire burns continuously for six days and nights, reaching temperatures around 1000°C at the end of the burning

period. After firing, the bricks cool in the kiln for about one week. About half of the bricks will be fired to the ideal hardness, and the remaining half will be either underfired or clinkers, depending on their proximity to the fires.[96]

The bricks utilized in this research were placed far away from the fire in the kiln and, as a result, did not reach the maximum temperature. These bricks were fired at approximately 950°C, which is the upper limit of what has been established to be the ideal temperature for pozzolanic reactions to occur. XRD showed that the minerals were quartz (SiO_2) and microline, a type of feldspar with the chemical formula $KAlSi_3O_8$. XRD did not yield conclusive results for the mineralogy of the bricks because of the difficulties associated with identifying clay minerals. Because clay minerals tend to be poorly-crystallized, they do not have typical patterns resulting from crystalline structures that are used to identify minerals in XRD. As discussed previously, at the temperature range at which these bricks were fired causes silica and alumina in the clays to lose their crystalline structures and become amorphous. XRD does not identify amorphous materials. XRD spectra can be found in Appendix C. The results of the other characterizations are displayed in the table 5.1.

[96] "Brickmakers," Colonial Williamsburg website, http://www.history.org/almanack/life/trades/tradebri.cfm, accessed March 23, 2011.

Table 5.1 Characterization of Williamsburg Brick Dust

Water Absorption	Acid Soluble Portion	pH	Nitrates	Chlorides	Sulfates	Munsell
49.3%	0.26%	7	none	none	0.5%	5 YR 6/6

5.2.3.2 BELDEN BRICK DUST

The second brick dust is from Belden Brick Company in Canton, Ohio. The bricks were produced in November, 2011. Belden Brick Company uses a modern shuttle kiln, but they were able to create a low-fired brick custom made for this research by controlling the temperature that the bricks reached in the kiln. The clay was first fully dried and then fired in a shuttle kiln. The bricks were removed from the kiln before reaching the temperature of 815°C. The fireclay is known as Brookville Clay and is considered to be a coal formation clay. X-Ray fluorescence oxide analysis provided by Belden Brick Company shows that the major components are silica (about 26-27%) and alumina (58-59%). XRD identified quartz, microline, and dehydroxylated muscovite. Dehydroxylated muscovite is a silicate mineral also known as common mica that has undergone deyhyroxylation through the firing of clay. Its chemical formula is $KAl_3Si_3O_{11}$. Again, clay minerals were not identified because of their poorly-crystallized or amorphous structure. The Belden brick dust contained less feldspar than the Williamsburg brick dust. XRD spectra can be found in Appendix C. The characterization of the Belden brick dust is displayed in Table 5.2.

Table 5.2 Characterization of Belden Brick Dust

Water Absorption	Acid Soluble Portion	pH	Nitrates	Chlorides	Sulfates	Munsell
54.1%	0%	7	none	none	0.5%	10 YR 8/1

5.3 FORMULATIONS

Two of the tests in the testing program (lime-pozzolan strength development and set time) required mortar formulations with established proportions of lime, sand, and additive (brick dust and marble dust). The proportions used in these formulations were derived from past research. The Smeaton project found that "in mortars based on non-hydraulic limes, the best performers were lime:sand:brick dust in proportions 1:3:1."[97] These proportions were used for the three formulations. Table 5.3 lists the formulations used in the current research. The amount of mixing water varied among the mixes depending upon the type of additive used. Approximate water requirements were determined before the mixing of formulations through preliminary tests in which mortars were mixed by hand to the appropriate consistency to which they could remain on an inverted trowel, a common test used in the field. Each mortar formulation was also subjected to the slump test using a flow table to ensure that

[97] Jean Marie Teutonico, Colin Burns, John Ashurst, and Iaian McCaig, *The Smeaton Project: Factors Affecting Lime-based Mortars*, APT Bulletin, Vol. 25, No. 3/4. (1993), p. 35.

the flow was reasonably consistent among all three mortars. One batch was made of each formulation which provided all specimens for lime-pozzolan strength development and set time tests.

Table 5.3 Proportions of Components by Volume

Formulation	Hydrated High Calcium Lime	Bar Sand	Williamsburg brick dust	Belden brick dust	Marble dust
A	1	3	1	--	--
B	1	3	--	1	--
C	1	3	--	--	1

5.4 PREPARATION OF SAMPLES

All samples were prepared in the Architectural Conservation Laboratory during the months of March and April, 2011.

5.4.1 MIXING

Specimens used in lime-pozzolan strength development and set time tests were mixed according to ASTM C 305 *Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency.* Slight modifications were made because of the differences in consistency and mixing requirements for lime mortar and hydraulic cement. The mechanical mixer used for mixing was a Hobart C-100 mixer with three speeds. The temperature of the laboratory at the time of mixing was 22°C and the relative humidity was 25%. The dry paddle and dry bowl were first placed in the mixing position in the mixer.

Approximately half of the deionized mixing water was added to the bowl. The hydrated lime and pozzolan, previously proportioned, blended together by hand, and stored in a closed container, were then added to the water in the mixing bowl and allowed to stand for one minute. The mixer was started at a slow speed for 30 seconds. After 30 seconds, the entire quantity of sand was slowly added while maintaining a slow speed of mixing. After four minutes of mixing, the remainder of the water was added to the mix. At five minutes, the mixer was stopped and the sides of the bowl were scraped down using a plastic spatula for 1 ½ minutes. Mixing was then resumed on medium speed for 2 ½ minutes, stopping to scrape the sides once again before completing mixing on medium for an additional minute. After mixing, the mortar was emptied into a mortar mixing pan where it continued to be mixed by hand using trowels for an additional 20 minutes before molding. While hand-mixing, additional water was incorporated into the mix as needed using a spray bottle with a premeasured amount of deionized water. Hand mixing was necessary because the mechanical mixing was not sufficient to fully integrate the binder, aggregate, and water to reach an appropriate mortar consistency.

5.4.2 MOLDING

Specimens for lime-pozzolan strength development and set time tests required the use of two different sample shapes. The compressive strength test required 2 inch cube molds that complied with ASTM 109 *Standard Test Method*

for Compressive Strength of Hydraulic Cement Mortars. These molds were custom-made, built of wood with three tightly-fitted cube compartments per mold, fitted together with wood screws, and plane surfaces on interior faces. The set time test required a ring mold with a base diameter of 70 mm, top diameter of 60 mm, and height of 40 mm and made of noncorroding, nonabsorbent material. The wooden molds were cleaned and coated with mineral oil while the set time cylinders were coated with petroleum jelly.

Wood molds overfilled with fresh mortar and continuously compacted with a putty knife to minimize voids. The tops of the overfilled molds were smoothed with a trowel and then the excess was sliced off with a metal putty knife in one fluid motion. Conical molds for set time tests were filled by hand by pressing a ball of mortar with the palm of the hand into the larger end of the mold and then slicing the top of the mold off in one fluid motion with a metal putty knife. Although not included in this testing program, molds for water vapor transmission were filled for potential future phases of this project. These molds are small cylinders ½ inch high and 1 ½ inches in diameter constructed of PVC pipe. They were overfilled by hand and the tops were cut off using a small putty knife.

Table 5.4 Sample Mold Schedule

Test	Standard	Mold Shape	Mold Size	Samples per Formulation	Total
Lime-pozzolan strength development	ASTM 109	Cube	2 in.	15	45
Set Time	ASTM 191	Truncated cone	70 mm base diameter, 60 mm top diameter, 40 mm height	3	9
Water vapor transmission	ASTM E 96	Cylinder	1 ½ in. diameter ½ in. height	6	18

5.4.3 CURING

The lime-pozzolan strength development test and the set time test both required specified curing conditions. According to ASTM 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime*, lime-pozzolan mortars should be cured in high temperatures and high relative humidity. The standard requires the use of a vapor oven at 130ºF for 7 days followed by curing at 73ºF at 95-100% relative humidity. This was not possible because the lab was not equipped with a vapor oven. Instead, the specifications in ASTM 109 *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars* were used for curing of compressive strength cubes and water vapor transmission cylinders. A moist cabinet was created using a bakers' rack

equipped with a plastic tent cover with a zipper. The only point of air entry was at the bottom of the rack where it was open. Five trays of water were set on the top, bottom, and various racks between sample trays. A dial hygrometer was hung with wire at the top of the bakers' rack to monitor temperature and relative humidity. Temperature ranged from 62°F to 71°F and relative humidity from 65% to 98% during curing. The samples were removed from the wooden molds at 8 days then returned to the moist cabinet until the time of compressive strength testing at 30 days.

ASTM 191 *Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle* requires that samples be stored in a moist cabinet with a temperature of 73.4ºF and a relative humidity no less than 90% in between readings. A moist chamber was created using a plastic container with a tightly-fitting lid measuring about 1 ½ feet by 2 feet. Four Petri dishes of water were placed inside amongst the samples. A digital hygrometer was placed inside to measure temperature and relative humidity over the duration of the testing and the lid was tightly closed. Temperature ranged from 17.5°C to 22.5°C and relative humidity ranged from 73% to 94%.

Figure 5.1: Reducing particle size of brick using ball mill (Rogers)

Figure 5.2: ASTM No. 200 sieve (Rogers)

Figure 5.3: Williamsburg brick dust (Rogers)

Figure 5.4: Belden brick dust (Rogers)

Figure 5.5: Molds and tools prepared for mixing and molding of mortar samples (Rogers)

Figure 5.6: Mixing Formulation B using Hobart C-100 mixer (Rogers)

Figure 5.7: Molding Formulation B for slump test (Rogers)

Figure 5.8: Moist cabinet for curing of mortar samples (Rogers)

CHAPTER 6 TESTING PROGRAM

6.1 INTRODUCTION

The testing program includes a variety of tests that address pozzolanicity indirectly through examination of physical properties (mechanical strength and set time) and chemical properties (lime absorption). The testing program was designed after a literature review of current and historic literature about pozzolans. The tests that were selected are from various sources and, while some have current standards (ASTM), others were derived from historical literature with only brief descriptions of the test procedure, which often varied from source to source. For the tests that do have modern standards, these were followed as closely as possible and adapted where necessary to be more applicable to lime mortars. For tests that were derived for historic literature, the test procedures were ultimately designed by interpreting the original sources and some trial and error preliminary tests to determine the most effective way to perform the test.

6.2 DETERMINATION OF FLOW

The flow of mortar formulations was determined not as a measure of pozzolanicity but, rather, to ensure a uniform consistency among samples dependent on the water requirement. Flow was determined according to ASTM C1437 *Standard Test Method for Flow of Hydraulic Cement Mortar* with one

slight modification in accordance with ASTM 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime.* It was performed immediately after the conclusion of hand-mixing and before the molding of specimens for set time tests. For this test, a flow table was used that was built for the Architectural Conservation Lab to conform with ASTM C230 *Standard Specification for Flow Table for Use in Tests of Hydraulic Cement*. The flow table is constructed of a plywood table top covered with a ¼ inch thick piece of plexiglass. The base is constructed of plywood as well. Underneath the plexiglass is a piece of paper with eight equidistant lines drawn across the top for taking flow measurements. A pipe is screwed into a 1 inch flange that is attached to the bottom of the plywood table top. A pipe 1¼ inches in diameter by 5 inches in length is screwed into a 1¼ inch flange attached to the base of the table. The smaller pipe fits inside the larger pipe allowing the table to be moved up and down.

The flow table was mounted securely to the laboratory table top using two clamps. The top of the flow table was wiped clean and dry and a flow mold was placed at the center. The flow mold conformed to ASTM 230 *Standard Specification for Flow Table for Use in Tests of Hydraulic Cement*: a conical, bronze mold with a base diameter of 4 inches, a top diameter of 2 ¾ inches, and a height of 2 inches. The mold was placed in the center of the flow table and filled with freshly-mixed mortar. The mortar was cut to a plane surface flush with the top of the mold by drawing the straight edge of a trowel with a sawing motion across the top of the mold. The table top was wiped dry and clean and any water around the edge of the flow mold was removed. After one minute, the mold was

lifted away and the table was immediately dropped a height of ½ inch 10 times in 6 seconds. While the flow test for hydraulic cement specifies that the flow table be dropped 25 times in 15 seconds, ASTM 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime* specifies, instead, 10 drops in 6 seconds because of differences in the consistency of lime mortars and hydraulic cement. The flow was determined by measuring the diameter of the mortar along the lines drawn on the table using digital calipers. This allowed the calculation of the percent increase of the original diameter of the mortar. The flow test was performed on two samples from each formulation

6.3 SETTING TIME TEST

The purpose of the setting time test is to measure initial and final set of mortar specimens as an indication of pozzolanicity in the relative rapidity of set. ASTM C191 *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle* was followed for the testing procedure. By means of a Vicat apparatus, the measurement of the penetration of the needle at various time intervals indicates the time of initial and final set, and the effects of the two brick dusts on set time are compared to each other and to the control.

This test was performed using mortar from the same batch as that used for lime-pozzolan strength development tests. After the completion of mixing and the flow test, the mortar was quickly molded into a ball with gloved hands and

tossed back and forth six times, maintaining the hands about 6 inches apart. The ball was then pressed into the larger end of the conical ring, completely filling the ring with mortar. The excess mortar was then removed from the bottom of the cone with a single movement of the palm of the hand. The filled mold was then placed, large end down, on a Plexiglass plate. The excess paste at the smaller top of the ring was removed by a single oblique stroke of a sharp-edged trowel held at a slight angle with the top of the ring. The top was smoothed with one or two light touches of the pointed end of the trowel. The specimen, sitting atop the plexiglass plate, was immediately placed in the 90% relative humidity chamber where it remained for the duration of the test with the exception of taking readings.

The time intervals for taking readings were established previously through preliminary tests, and each formulation had a different interval based on the amount of time predetermined for cure. The procedure for taking penetration readings was to lower the rod of the 1 mm Vicat needle until it rested on the surface of the mortar specimen. The set screw was tightened and the indicator was set to zero at the upper end of the scale. The rod was quickly released by releasing the set screw and the needle was allowed to settle for 30 seconds before the reading was taken to determine penetration. Initial set is considered to have occurred when the needle does not penetrate more than 25 mm into the mortar. The final setting is considered to have occurred when the needle does not visibly sink into the paste. Care was taken to ensure that no penetration test

was made closer than 3/8 inches from the inside of the mold and no closer than 1/4 inch to a previous test.

6.4 SET UNDER WATER TEST

The purpose of this test is to determine ability of a mortar paste to set under water as an indication of pozzolanic activity, as pozzolans are known to impart hydraulic properties to lime mortars allowing them to set under water without access to carbon dioxide. To the author's knowledge, there is no modern standard that exists for the test. It was developed by the French chemist Feret in 1925 and recommended in a paper by Moran and Gillian in the 1949 ASTM publication *Symposium on Pozzolanic Materials in Mortars and Concretes.* Using this source, along with ASTM C191 *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle*, the following procedure was developed for this testing program.

The Feret method described in *Symposium on Pozzolanic Materials in Mortars and Concretes* requires that the test be performed on a paste of 4:1 pozzolan: hydrated lime "gauged with sufficient water and mixed by hand to produce a paste of normal consistency." The 4:1 ratio is by weight rather than volume. The correct proportions of dry ingredients were weighed and combined in a bucket. The lime and brick dust were simply mixed by hand in a bucket with a trowel using enough water to form a paste consistency, recording the amount

of water used and ensuring comparable consistencies amongst the different formulations visually. The paste was then placed in a cylindrical glass jar and compacted by hand to ensure that there were no air bubbles and that the surface was relatively flat. The paste was then covered with 100 mL of limewater followed by a film of mineral oil to prevent the ingress of carbon dioxide or evaporation. Lime water was created by covering hydrated lime with about three inches of deionized water and allowing it to sit for one month. Glass jars were sealed with their plastic lids and the samples were stored in the laboratory at room temperature.

Following mixing and molding, ASTM 191 *Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle* was followed for performing the Vicat set time test. Samples were left in the lab with the lid of the jar sealed except during readings. The penetration of the 1 millimeter needle was determined every 24 hours until initial and final set was reached. According to Feret, the degree of activity of a pozzolan can be generally assessed based on the following: Active pozzolans show initial set in less than 50 hours and final set in less than 100. Poor or intermediate materials range from these values to no set. Inactive materials will not set. This test was performed on 3 samples for each additive.

6.5 LIME COMBINATION TEST

The purpose of the lime combination test is to determine if a material possesses pozzolanic potential and to measure its relative activity through a simple chemical field test. If a material is pozzolanic, it will form hydrated calcium alumino-silicates when mixed with lime and water. These C-S-H compounds are the reaction products that are responsible for the hydraulic properties of pozzolanic mortars. The formation of these compounds will result in an increase in solid matter that can be visually observed and approximately measured in a test tube or another container. Another indication of the formation of hydrated calcium alumino-silicates will be the slowed rate of settlement of solid matter. To the author's knowledge, there is no modern standard that exists for this test. It appears in A. D. Cowper's *Lime and Lime Mortars* in 1927. He refers to the test as a "Practical Test for Pozzolanic Properties."[98]

Hydrated lime, brick dust, and distilled water were added to a 50 mL graduated cylinder in the following proportions: 0.5 g brick dust, 0.3 g hydrated lime, and 20 mL distilled water. The graduated cylinders were stoppered with rubber stoppers then sealed further with the application of parafilm, a paraffin film, and wrapped with electrical tape around the mouth of the graduated cylinder. Specimens were stored in the laboratory in room temperature. After 12 hours, each graduated cylinder was shaken vigorously for 10 seconds. At the time of shaking, the volume of solid matter was noted and recorded every 2 and

[98] A.D. Cowper, *Lime and Lime Mortars,* Shaftsbury, UK: Donhead Publishing Co., 1927, pp. 48-49.

4 minutes by observing the level of solid particles in the cylinder as they settled. This procedure was repeated every 12 hours for seven days. Five tests were performed for each brick dust. Five tests were also performed using marble dust as a control.

6.6 LIME-POZZOLAN STRENGTH DEVELOPMENT

The purpose of the lime-pozzolan strength development test was to measure the strength enhancement imparted to a lime mortar by the addition of crushed brick, as compared to a control specimen. The test used a combination of two ASTM standards. Section 9 *(Lime-Pozzolan Strength Development)* of ASTM 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime* establishes the procedure of using compressive strength testing to assess pozzolans and gives a minimum compressive strength of 600 psi at 28 days for pozzolans. This number, however, should not be used as a lower limit for pozzolanicity. ASTM 593 is a specification for uniformity in construction materials and may not be appropriate for conservation mortars which may not require such great strength. Therefore, any lime mortar that does not reach 600 psi at 28 days with the addition of brick dust should not be classified as non-pozzolanic. ASTM 593 includes compressive strength testing as one of six test methods for assessing pozzolans. It specifies adherence to another standard, ASTM 109 *Standard Test Method for Compressive Strength of Hydraulic Cement Mortars,* for mixing, molding, and testing mortar formulations in compression.

After the mixing of specimens in accordance with ASTM 305 *Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency* and measuring the flow using a flow table, the samples were molded into two inch wooden cube molds. Following molding, samples were placed in a moist cabinet for curing at around 90% relative humidity. They were removed from their molds after seven days then returned to the moist cabinet until the time of the compressive strength test at 30 days. In preparation for the test, each specimen was brushed and wiped clean to remove any loose sand grains or incrustations from the faces. The faces were checked for levelness and the surface area of the testing face was calculated by measuring with digital calipers.

While the standard on which this test was based (ASTM 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime*) requires testing of compressive strength at exactly 28 days to establish compressive strength of lime-pozzolan mortars, these samples were tested at 30 days instead for convenience. This small variation of time was inconsequential because all samples were tested after exactly 30 days of cure. Samples were tested in compression at the Laboratory for Research on the Structure of Matter at the University of Pennsylvania using an Instron Model 4206 static testing machine with the assistance of Dr. Alex Radin. The mortar cubes were placed with only true plane surfaces in contact with the bearing blocks of the machine, as specified by ASTM 109 *Standard Test Method for Compressive Strength of*

Hydraulic Cement Mortars. Constant force was applied from above until the mortar cube reached its point of failure due to stresses. The amount of force and displacement over time were recorded, as well as the point at which the mortar sample failed. This data was used to calculate compressive strength.

Figure 6.1: Setting time test samples in (uncovered) humidity chamber (Rogers)

Figure 6.2: Setting time measurement using Vicat apparatus (Rogers)

Figure 6.3: Handmixing lime and Williamsburg brick dust for underwater set test (Rogers)

Figure 6.4: Formulation A pastes molded into glass jars before submerging in water for underwater set test (Rogers)

Figure 6.5: Underwater set test, Formulation A (Rogers)

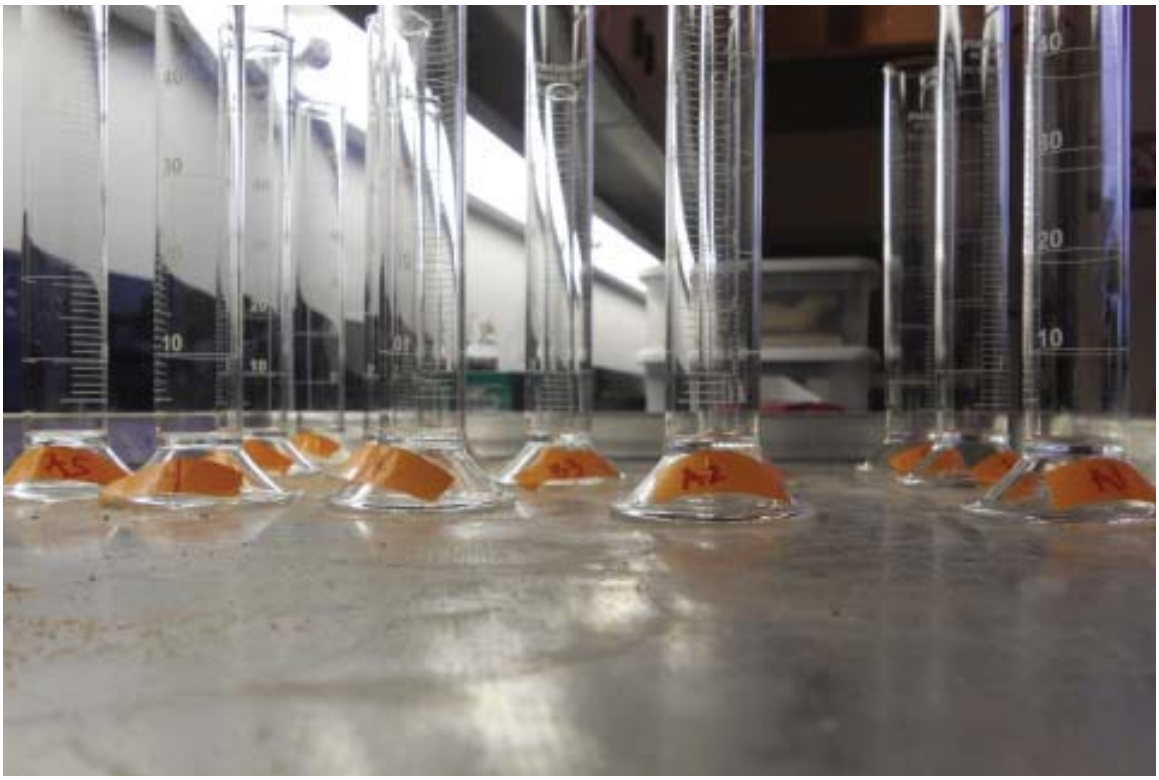


Figure 6.6: 50 mL graduated cylinders for Lime Combination Test (Rogers)

Figure 6.7: Preparation for Lime Combination Test (Rogers)

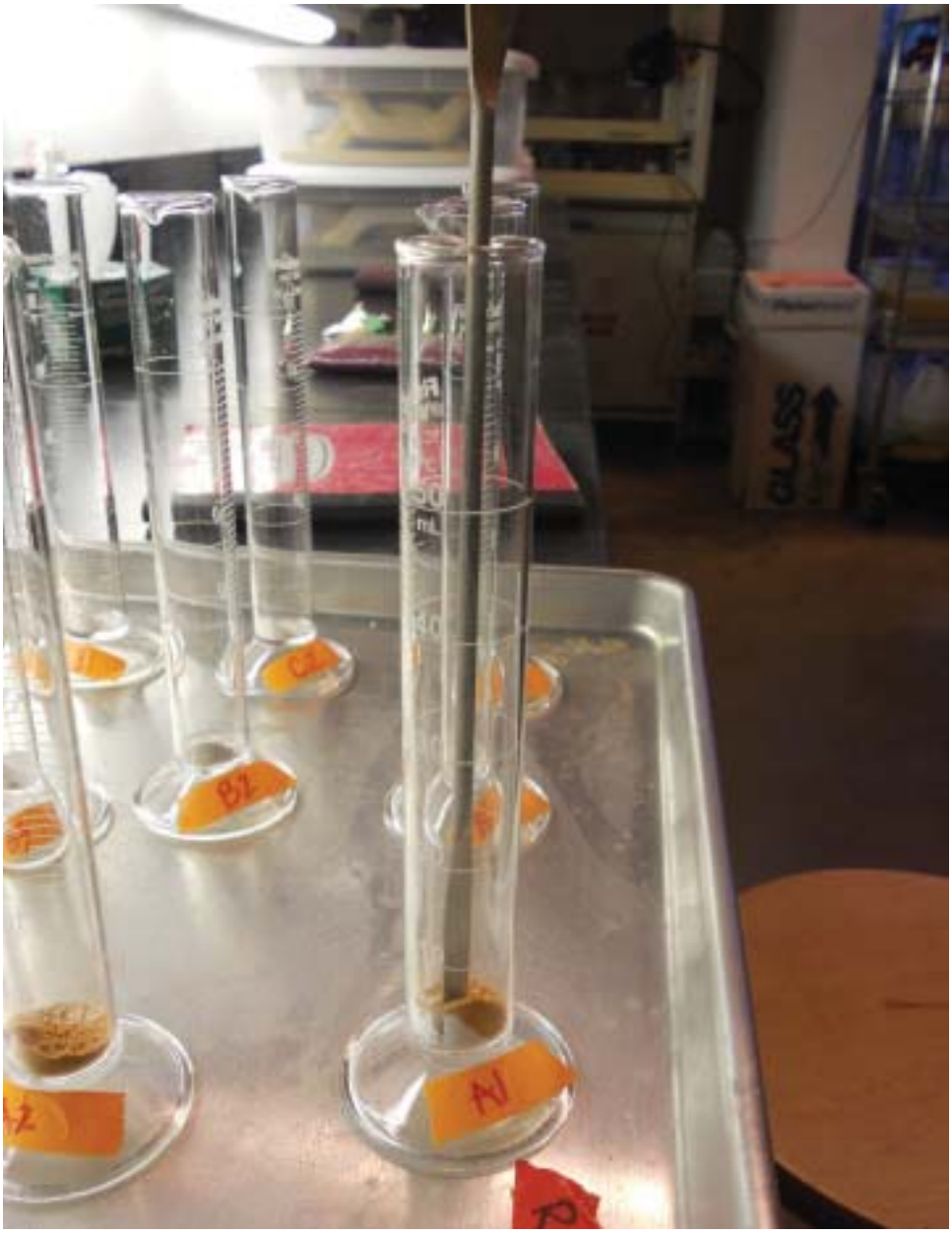


Figure 6.8: Mixing of lime and brick dust for Lime Combination Test (Rogers)

Figure 6.9: Applying Parafilm to seal cylinders (Rogers)

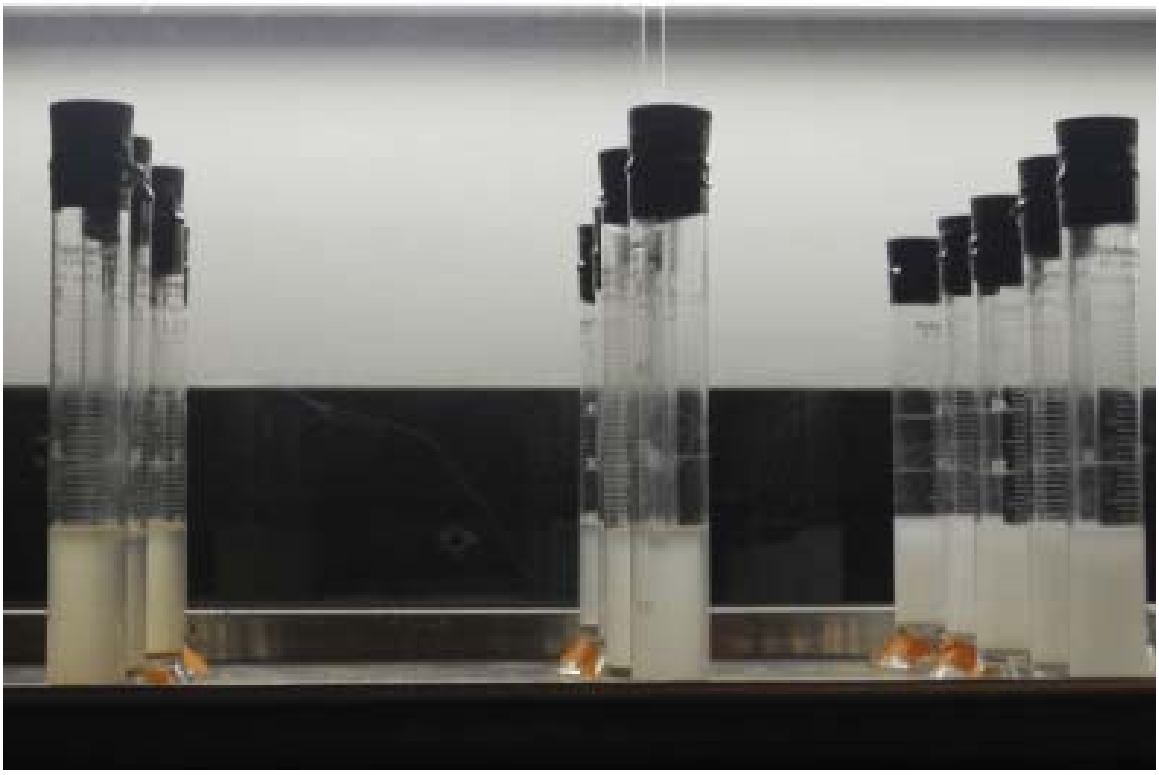


Figure 6.10: Lime Combination Test samples (Rogers)

Figure 6.11: Compressive strength sample mold (Rogers)

Figure 6.12: Compressive strength test samples (Rogers)

CHAPTER 7 TEST RESULTS

7.1 INTRODUCTION

All tests prescribed in the Testing Program chapter were performed in the Architectural Conservation Laboratory and the Laboratory for Research on the Structure of Matter at University of Pennsylvania during the months of March and April, 2011. The tests results are generally described below and corresponding appendices provide complete data resulting from the tests. The analysis of the efficacy of the tests the implications for field testing will be discussed in the following chapter.

7.2 DETERMINATION OF FLOW

The flow test was performed on each mortar formulation to be used for the lime-pozzolan strength development and setting time tests for the purpose of ensuring a similar consistency among the different formulations dependent upon the water requirement. It should be classified separately from those tests performed for pozzolanicity evaluation, as it is only used in this testing program as a measure of consistency among batches. ASTM C1437 *Standard Test Method for Flow of Hydraulic Cement Mortar* was followed as guide for determining flow with the flow table method. Flow is the increase in the average base diameter of a mortar mass expressed as a percentage of the original base diameter. It is calculated from the following equation:

$$F= A-B / B \times 100$$

where:

F= percent flow

A= average of four readings in millimeters,

B= original inside base diameter in millimeters

The results of flow tests are presented in Table 7.1 and complete data is recorded in Appendix E. ASTM C1437 *Standard Test Method for Flow of Hydraulic Cement Mortar* specifies a standard deviation of 4 and a difference of no more than 11% flow between two tests for each batch performed by a single operator in a single laboratory. The flow measurements do meet those requirements for precision.

Table 7.1 Flow Test Results

Formulation	Average Base Diameter (mm)	% Flow	Average % Flow
A1	112.5	10.7	
A2	112.3	10.6	10.7
B1	108.2	6.5	
B2	106.5	4.9	5.7
C1	107.2	5.6	
C2	107.2	5.5	5.6

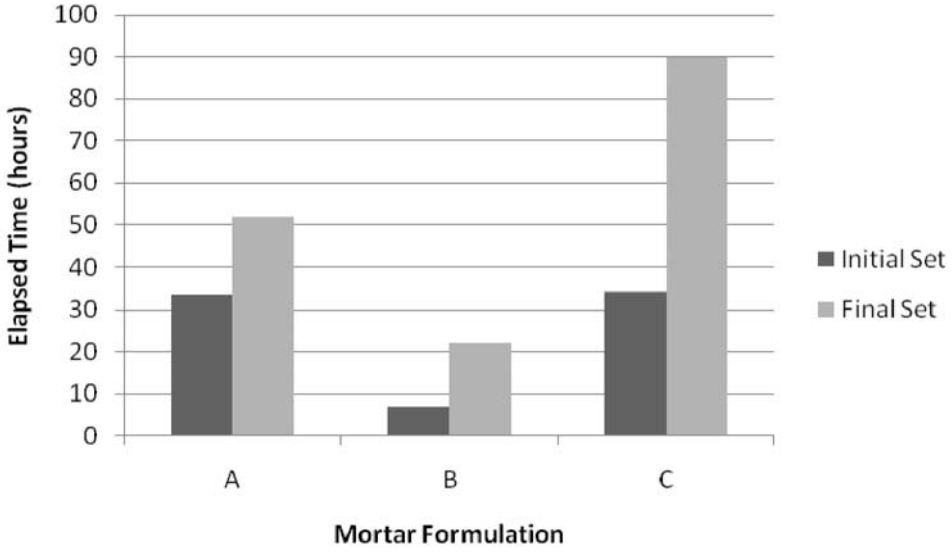
7.3 SETTING TIME

The setting time of mortar specimens was determined according to ASTM C191 *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle* in order to examine the effects of brick dust on rate of curing and assess the test's ability to predict pozzolanicity. Both initial and final set were recorded. Formulation B, containing the Belden brick dust, set the fastest reaching final set at 22 hours. Formulation A, containing the Williamsburg brick dust, followed reaching final set at 52 hours. Formulation C, the control, was the slowest to set requiring nearly 4 days (90 hours) to reach final set. Complete data from setting time tests can be found in Appendix F. Table 7.2 and Graph 7.1 display average results from setting time tests.

Table 7.2 Setting Time Test Results

Formulation	Average Initial Set (hours)	Average Final Set (hours)
A	33.5	52
B	7	22
C	33	90

Graph 7.1 Setting Time Test Results



Sample Key	
Formulation A	Williamsburg Brick Dust
Formulation B	Belden Brick Dust
Formulation C	Marble Dust Control

Samples for this test contain 1:3:1 lime: sand: additive

7.4 SET UNDER WATER TEST

The ability of lime and brick dust pastes to set under water was tested in order to measure pozzolanicity based on the property of lime-pozzolan composites to set in water without access to carbon dioxide. The paste formulations were created according to a method proposed by Feret[99] and setting

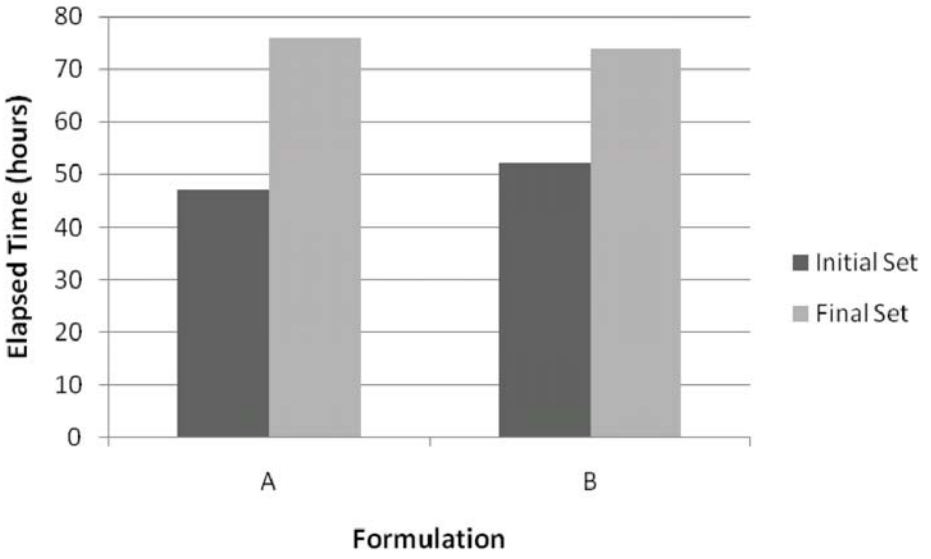
[99] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 116.

time was measured using ASTM C191 *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle*. The test demonstrated that both Brick Dust A and Brick dust B had the property of setting underwater. The control, however, showed no set at all, even after one week at which point the test was discontinued. Brick Dust B set the most rapidly, as it did in the setting time test. However, Brick Dust A began to set before Brick Dust B, and reached initial set several hours before Brick Dust B. Brick Dust A had a very rapid setting period between 48 and 60 hours, after which its rate of setting slowed. Brick Dust A and Brick Dust B set within only two hours of each other, reaching final set at 76 and 74 hours, respectively. According to Feret, both are within the range of active pozzolans, which show initial set in less than 50 hours and final set in less than 100 hours. Complete data from underwater set tests can be found in Appendix G. Table 7.3 and Graph 7.2 display average results from underwater set tests.

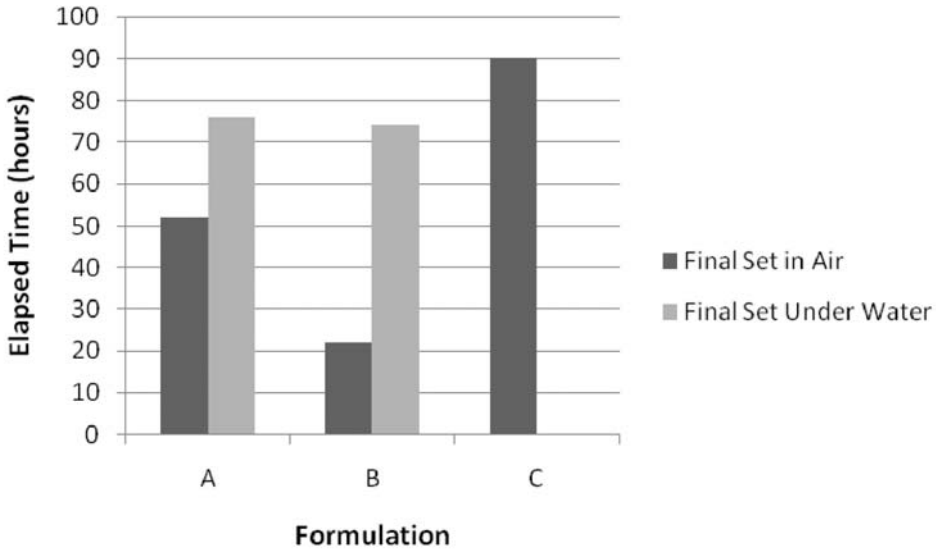
Table 7.3 Underwater Set Test Results

Formulation	Average Initial Set (hours)	Average Final Set (hours)
A	47	76
B	52	74
C	No set	No set

Graph 7.2 Underwater Set Results



Graph 7.3 Set Time in Air vs. Underwater Set Time



Sample Key	
Formulation A	Williamsburg Brick Dust
Formulation B	Belden Brick Dust
Formulation C	Marble Dust Control

Samples for this test contain 1:4 lime: additive

7.5 LIME COMBINATION TEST

The lime combination test measures the ability of a pozzolan to combine with lime to form calcium silicate hydrates. Data is obtained through visual observation of the volume of solid mass in a solution of lime and pozzolan. This is meant to be a simple field test that measures pozzolanicity on a comparative basis. Although the results were not very quantifiable, comparisons were made through measurements that can be found in Appendix H.

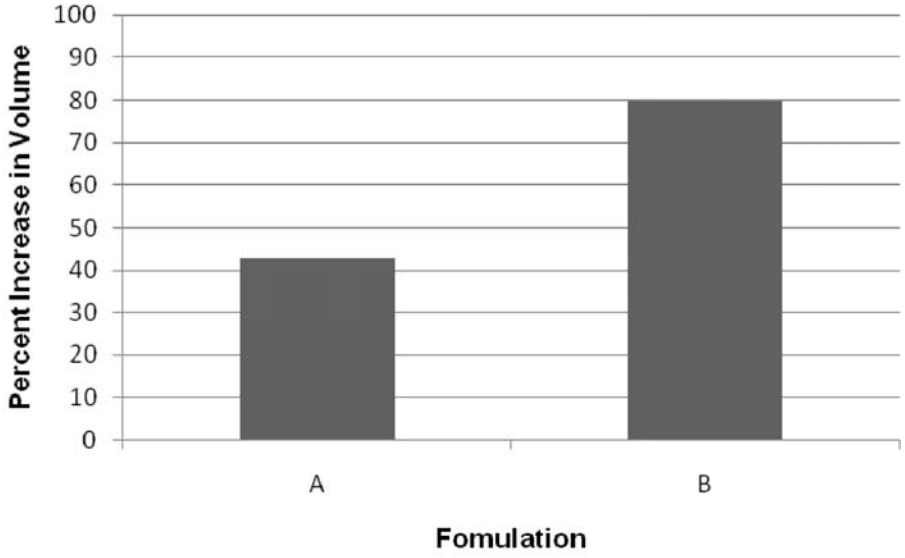
This test resulted in the reaction described by A.D. Cowper in his 1927 treatise on lime mortars. The formation of calcium silicate hydrates led to an increase in solid matter in the test tube that appeared as a flocculent substance of a consistency very different from the control sample. Over the course of seven days, the volume of solid material increased as the rate of settling after shaking slowed. The results were recorded by noting the height of solid matter based on 1 mL graduations on a 50 mL graduated cylinder as the suspension settled post-agitation.

The reaction was immediately noticeable in Brick Dust B (Belden) after the initial agitation. At the end of the 7-day testing period, the solid matter observed in this solution was nearly 5 times greater (80%) than it been at the initiation of the test. Brick Dust A (Williamsburg) had much less dramatic results. While an increase in solid matter was observed, it was slower and was not observed significantly until several days after the initiation of the test. The solid matter of Brick Dust A increased by 43 percent at the end of the 7 day testing period. The control sample experienced none of the changes observed in the pozzolan samples. At the end of the 7 day testing period, the amount of solid material was the same as it was at the beginning, and the consistency remained the same throughout (the material was not flocculent in appearance.) Upon shaking, the control samples formed a homogenous, milky solution that settled slowly preventing the reading of the level of solid matter at 2 minutes.

Table 7.4 Increase in Solid Volume due to Lime Combination

Formulation	Average Vol. Day 1 (mL)	Average Final Vol. (mL)	Increase (%)
A	2.0	3.5	42.9
B	1.8	8.9	79.8
C	1.0	1.0	0.0

Graph 7.4 Increase in Solid Volume due to Lime Combination



Sample Key	
Formulation A	Williamsburg Brick Dust
Formulation B	Belden Brick Dust
Formulation C	Marble Dust Control

*Samples for this test contain 1: 0.6 additive to lime.

7.6 LIME-POZZOLAN STRENGTH DEVELOPMENT

The lime-pozzolan strength development test measures compressive strength enhancement imparted to a mortar mix through the addition of a pozzolan. The equation for compressive strength is

$fm = P/A$

where:

fm = compressive strength in psi

P = total maximum load in lbs

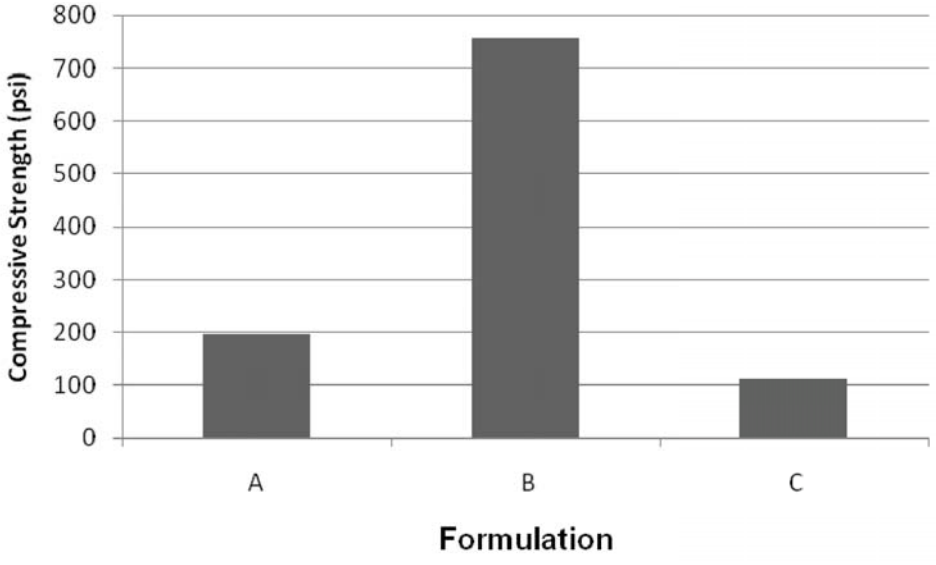
A= area of loaded surface in in^2

Formulation B, the mortar containing Belden brick dust, had significantly higher compressive strength than Formulations A or C. Formulation A, the Williamsburg brick dust, performed slightly better than the non-pozzolanic control. The strength of Formulation B was 523.8% higher than the non-pozzolanic control and the strength of Formulation A was 75% higher than the control. Table 7.4 provides the compressive strength, calculated as an average of the 5 samples.

Table 7.5 Compressive Strength

Formulation	Compressive Strength (psi)	Average Compressive Strength (psi)
A	223.57	197.56
	181.27	
	243.10	
	155.05	
	184.79	
B	--	756.72
	497.30	
	817.22	
	827.99	
	894.38	
C	63.15	112.92
	117.64	
	132.54	
	111.72	
	139.56	

Graph 7.5 Compressive Strength



Sample Key	
Formulation A	Williamsburg Brick Dust
Formulation B	Belden Brick Dust
Formulation C	Marble Dust Control

Samples for this test contain 1:3:1 lime: sand: additive

It should be noted that an oversight in testing procedure led to incomplete data in the results of Formulation B. The unexpectedly high strength displayed by Formulation B was not accounted for in the initial load range for which the data collector function was preset. It was set at a 2,000 pound limit, as was sufficient for Formulation A, and this limit was surpassed by sample B1 and the point of

failure was not recorded. As a result, sample B1 was omitted from the calculation of the average compressive strength. Because of the vastly different strengths of the three formulations, ranging from 112 psi to over 700 psi, the load parameters had to be changed for each formulation. Complete compressive strength data and graphs can be found in Appendix I.

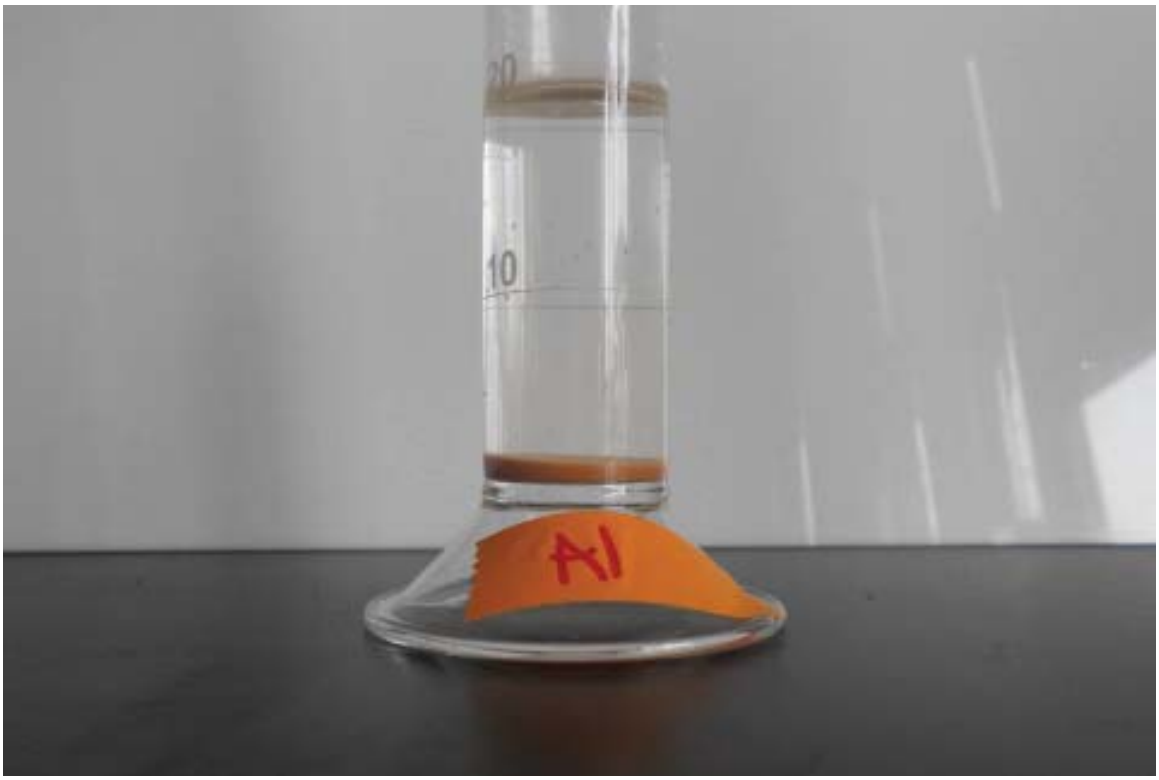


Figure 7.1: Sample A1, day 1, before agitating (Rogers)

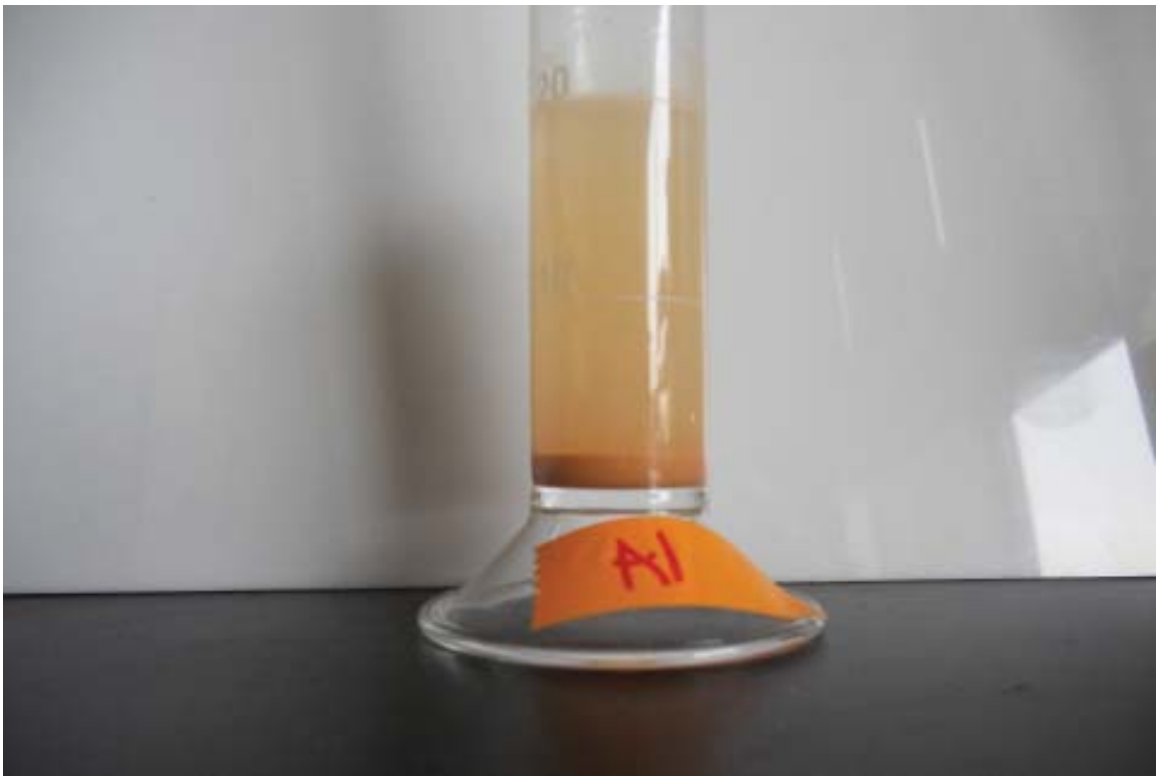


Figure 7.2: Sample A1, day 1, after agitating (Rogers)

Figure 7.3: Sample B1, day 1, before agitating (Rogers)

Figure 7.4: Sample B1, day 1, after agitating Rogers)

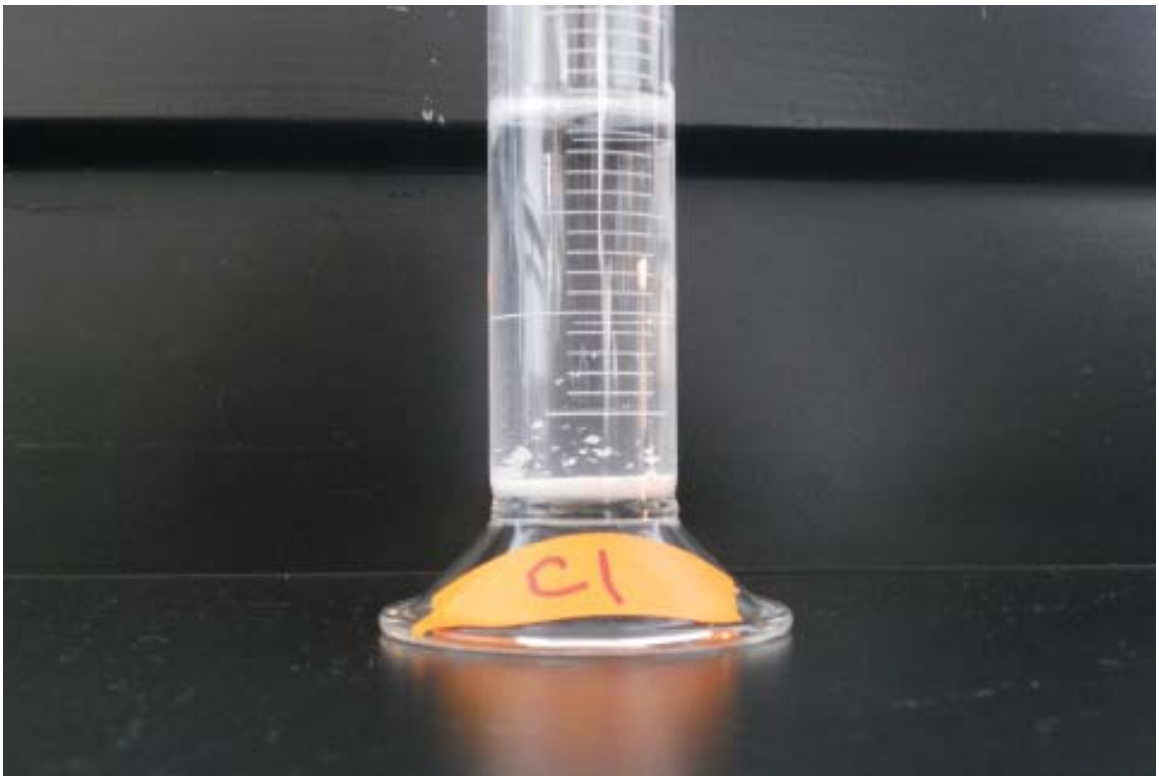


Figure 7.5: Sample C1, day 1, before agitating (Rogers)

Figure 7.6: Sample C1, day 1, after agitating (Rogers)

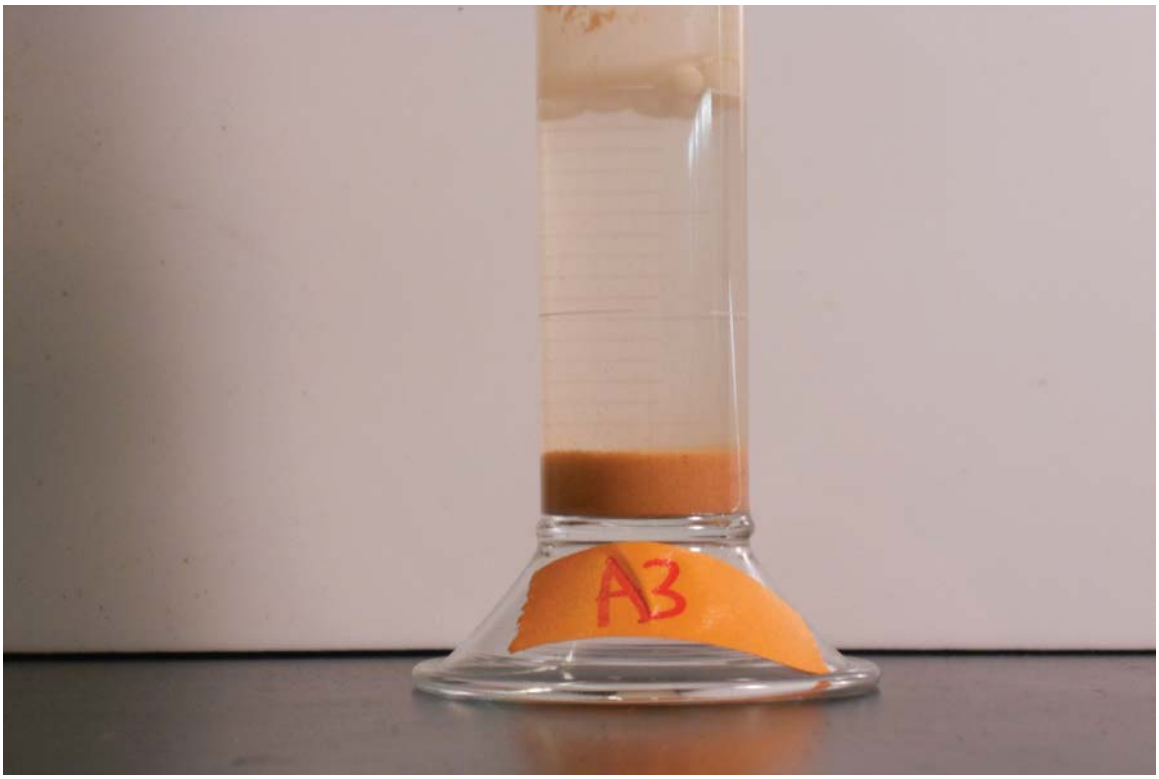


Figure 7.7 Sample A3, day 7, before agitating (Rogers)

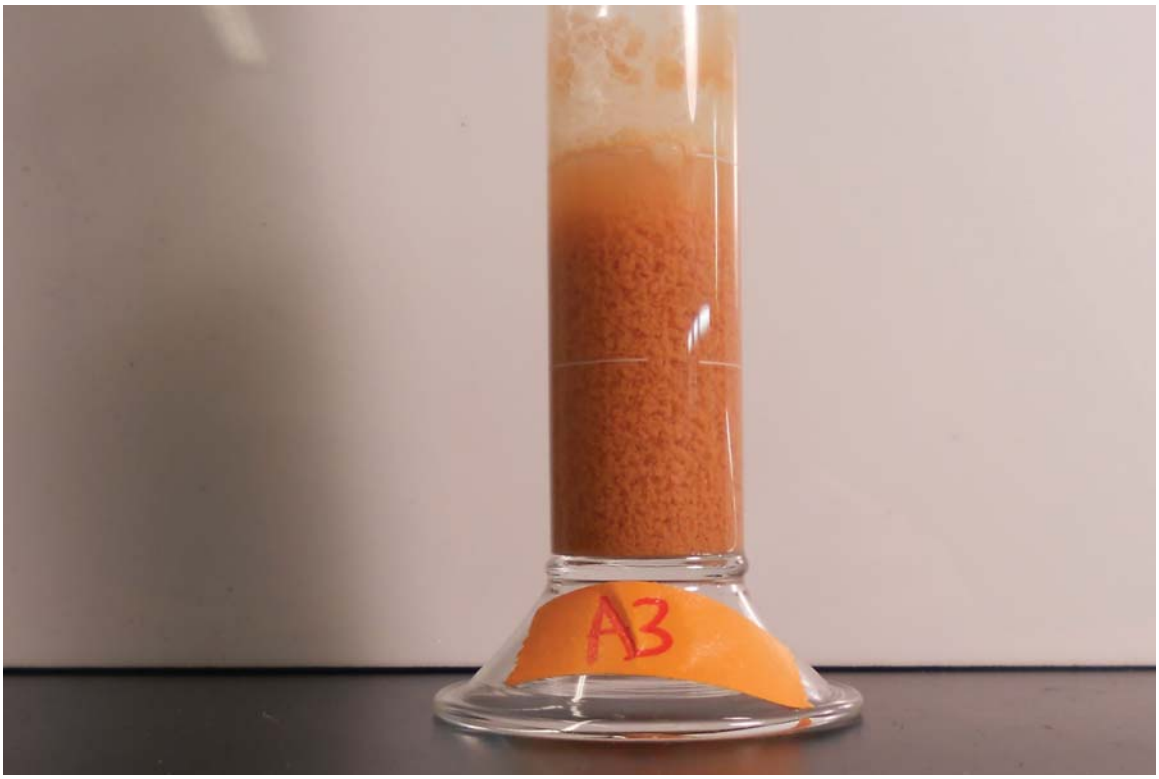


Figure 7.8: Sample A3, day 7, after agitating (Rogers)

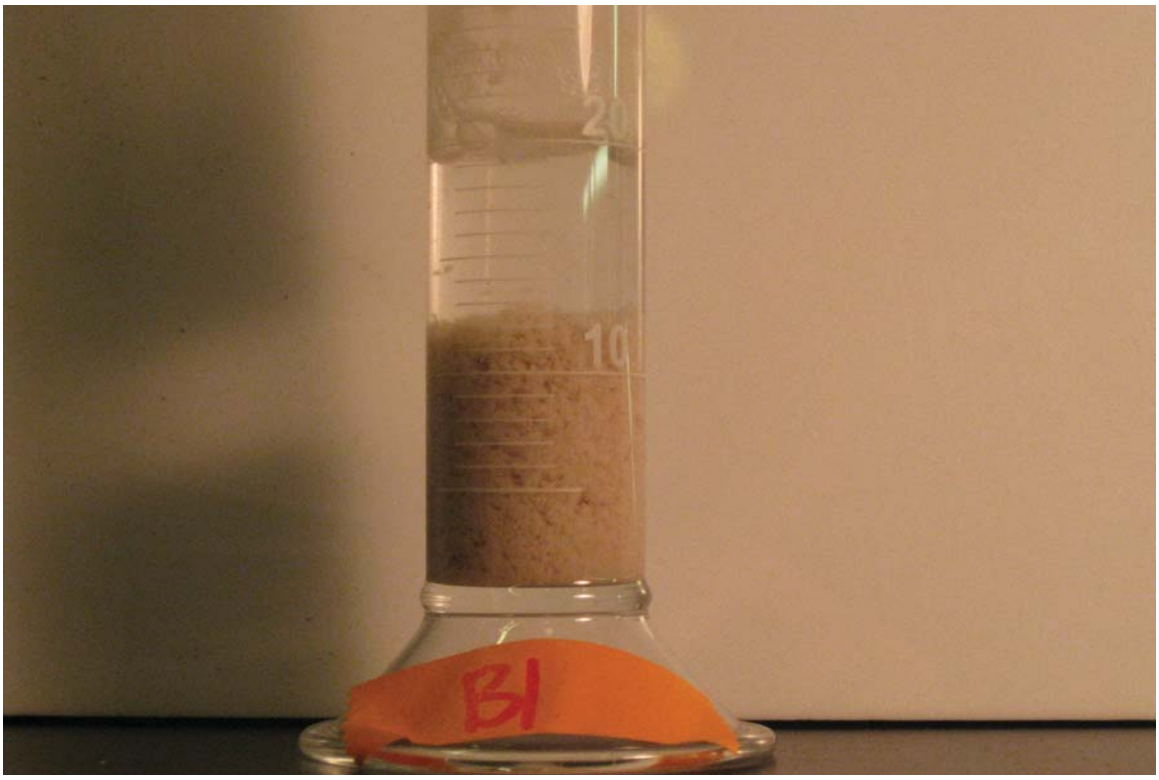


Figure 7.9: Sample B1, day 7, before agitating (Rogers)

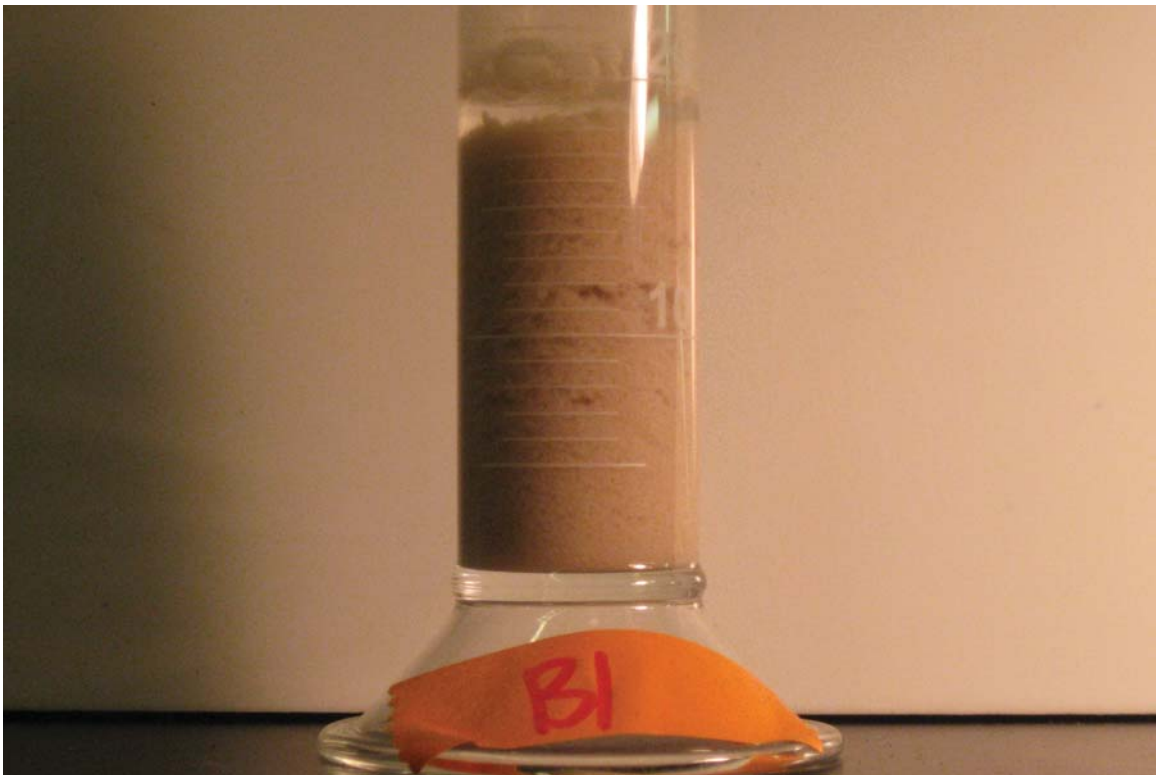


Figure 7.10: Sample B1, day 7, after agitating (Rogers)

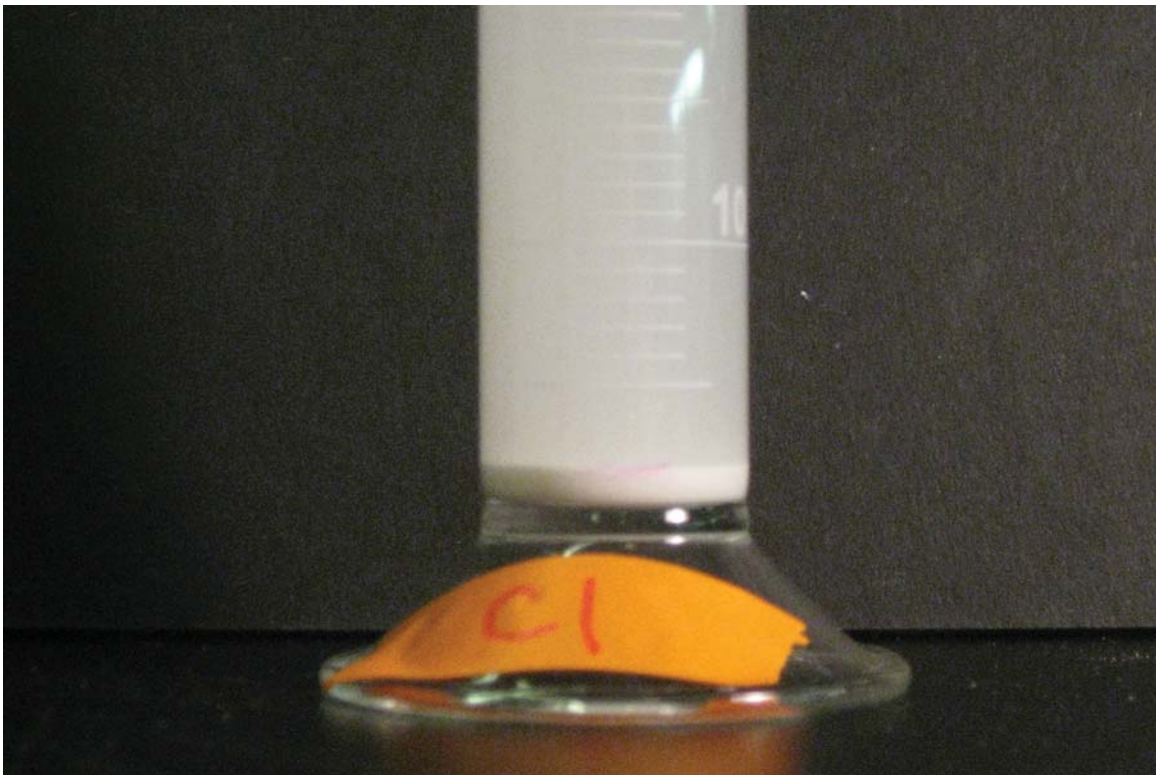


Figure 7.11: Sample C1, day 7, before agitating (Rogers)

Figure 7.12: Sample C1, day 7, after agitating (Rogers)

Figure 7.13: Compressive Strength Test using Instron Model 4206 at LRSM (Rogers)

Figure 7.14: Compressive Strength Test, Formulation A mortar cube before crushing (Rogers)

Figure 7.15: Compressive Strength Test, Formulation A mortar cube after crushing (Rogers)

Figure 7.16: Compressive Strength Test, Formulation B mortar cube before crushing (Rogers)

Figure 7.17: Compressive Strength Test, Formulation B mortar cube after crushing (Rogers)

Figure 7.18: Compressive Strength Test, Formulation C mortar cube after crushing (Rogers)

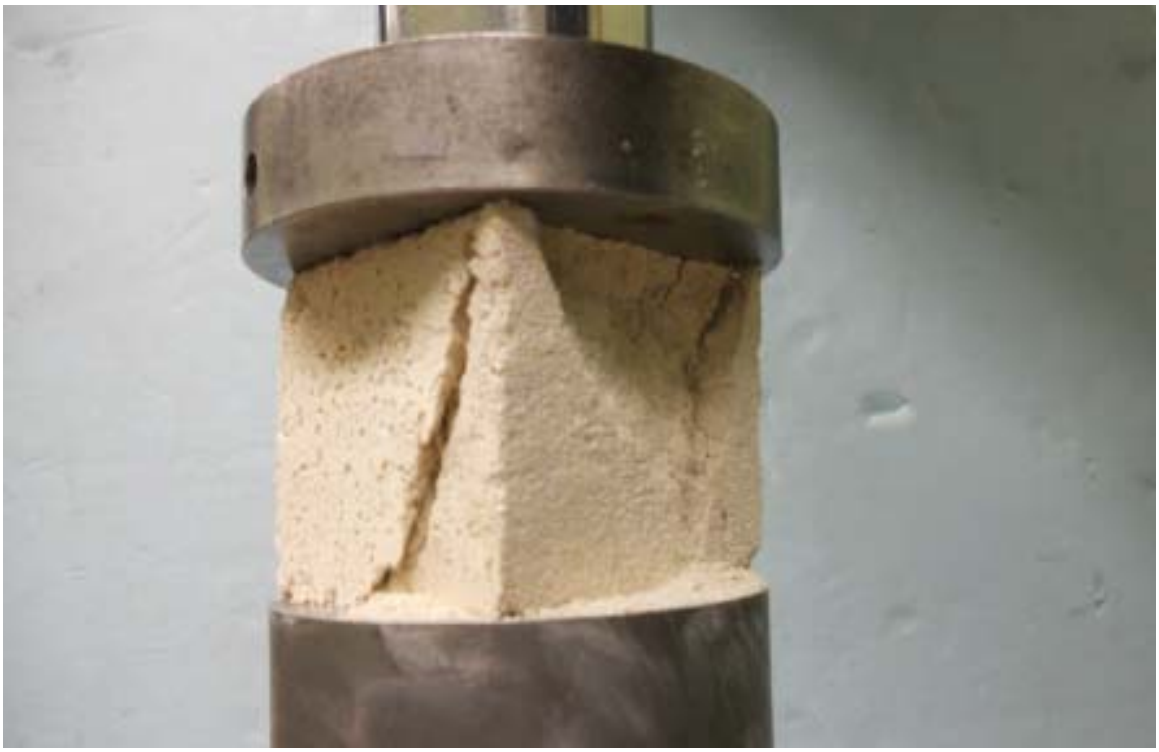


Figure 7.19: Compressive Strength Test, Formulation C mortar cube after crushing (Rogers)

CHAPTER 8 DISCUSSION OF RESULTS

8.1 INTRODUCTION

The pozzolanicity tests performed for this testing program provided different types of data pertaining to the effect of two different brick dusts on the properties of lime mortars. These results were interpreted to determine pozzolanicity. The test results will be discussed below, and conclusions about the efficacy of the tests will be drawn.

8.2 SETTING TIME TEST

The setting time test proved to be an effective method of evaluating pozzolanicity. The test measured pozzolanicity indirectly by examining the effects of brick dust on the amount of time it takes for a given formulation to achieve set. Pozzolans decrease set time in lime-based mortars because the reactive silica and alumina in the pozzolan fix calcium hydroxide in the formation of cementitious calcium silicate hydrates. The setting and curing action in pozzolanic mortars is accomplished through a combination of carbonation from carbon dioxide in the air and the formation of calcium silicate hydrates. These two reactions in concurrence will cause the mortar to set much faster than a mortar that sets through the single process of carbonation. This is consistent with the results of this test, as Formulation A, made with Williamsburg brick dust, set

42% faster than the non-pozzolanic control and Formulation B, made with Belden brick dust, set 76% faster than the control and twice as fast as Formulation A.

In addition to indicating positive pozzolanicity as compared to a non-pozzolanic control, this test also has the ability to measure relative degrees of pozzolanicity through the comparison of the results of different formulations of pozzolanic mortars. The difference between the setting times of Formulation A and Formulation B would suggest that Formulation A is less pozzolanic than Formulation B, meaning that it formed less calcium silicate hydrate resulting in a slower set. Without more in-depth material analysis of the samples, it cannot be confirmed that this decreased setting time directly correlates to the quantity of calcium silicate hydrate formed, but it is a reasonable assumption given that the only variable in the experiment was the brick dust itself. This difference between samples is valuable because it indicates a certain degree of precision with the test in differentiating between two brick sources and determining relative degrees of pozzolanicity

It should be noted that the curing conditions had a major influence on the results of this test. The samples were cured in a humidity chamber at 90% relative humidity, as per ASTM 191 *Standard Test Methods for Time of Setting of Hydraulic Cement by Vicat Needle*, in order to eliminate variables of air temperature and humidity. When mortars are used in the field, humidity and temperature during curing would be very different, and would fluctuate over the course of curing time.

A preliminary set time test was performed on the same mortar formulations in open air, at room temperature and relative humidity around 35-40%. The results of these tests are not reported because they were not performed to laboratory standards, but it can be stated that both brick dust mortars cured up to twice as fast as they did in the high humidity chamber. The results, however, were consistent with the results of the controlled cure in terms of the relative speeds at which the two mortars set, as compared to a control and to each other. Therefore, this test could reasonably be performed without controlling temperature and humidity, and would also be much faster, as high humidity slows curing time especially in non-pozzolanic control mortars.

8.3 SET UNDER WATER TEST

. The set under water set test is an effective method for determining pozzolanicity in the respect that, if performed correctly, it cannot possibly give a false positive. The test measures pozzolanicity indirectly by examining a property that brick dust is known to impart to mortar: hydraulicity, or the ability to set under water. A pure lime mortar with no pozzolanic additives will not, under any circumstances, set when submerged in water. A pure lime mortar that does contain a sufficient amount of pozzolanic additives will, theoretically, eventually set under water. As a caveat, using a pure lime is very important in this test because lime that has hydraulic properties will cause under water set and could potentially give a false positive for a non-pozzolanic material. The amount of time

required for the set to occur should provide some indication of the relative degree of pozzolanicity as a result of the amount of calcium silicate hydrate formed. According to Feret's classification, pozzolans can be broadly categorized based on the amount of time they require to set under water.[100]

Table 8.1 Pozzolanicity Classification of Sample Mortars Based on Feret's System for Set Time Under Water

	Active (final set in < 100 hours)	**Poor** (final set in >100 hours)	**Inactive** (no set)
Formulation A	76 hours		
Formulation B	74 hours		
Formulation C			No set

Sample Key	
Formulation A	Williamsburg Brick Dust
Formulation B	Belden Brick Dust
Formulation C	Marble Dust Control
Samples for this test contain 1:4 lime: additive	

The results of the test clearly confirm that the Williamsburg and Belden brick dusts are both active pozzolans because they set under water within 100 hours.

An unexpected outcome of this test, however, was the relationship between set time in air and set time under water. Because Formulation B set

[100] W. T. Moran and J. L. Gilliand, *Summary of Methods for Determining Pozzolanic Activity,* Symposium on Use of Pozzolanic Materials in Mortars and Concretes, ASTM Special Technical Publication No. 99, 1950, p. 116.

58% faster than Formulation A in humid air curing, it was hypothesized that Formulation B was more pozzolanic and would set significantly faster under water as well. However, Formulation B only set two hours faster under water than Formulation A (as opposed to 30 hours in air), and actually reached initial set 5 hours after Formulation A.

The narrow variation in final setting time between the two pozzolanic formulations suggests that this test is accurate but not particularly sensitive. In other words, it is accurate in providing a positive/negative indication of pozzolanicity but not precise in detecting the differences between the brick dust formulations that would make it capable of comparing pozzolanicity of different materials. However, for a simple determination of whether a material is pozzolanic, this test may be sufficient. The degree of sensitivity required for field testing is dependent upon the specifics of the project. This test may be suggested as a preliminary method of “weeding out” sources of brick dust that are clearly feeble inert. Then, should the project require more precise characterization of the material, other tests could be performed on brick dusts that passed the set under water test to determine its effects on other properties and its relative degree of pozzolanicity.

8.4 LIME COMBINATION TEST

The lime combination test was successful in identifying pozzolanic brick dust by visual observation of the formation of calcium silicate hydrate reaction

products through a simple chemical method. As Cowper suggested, this test was intended to serve as a quick field test. It did not result any kind of quantitative measurement that can be used to precisely establish a degree of pozzolanicity or compare pozzolans according to quantitative measurements as can be done with timed setting tests or strength tests. It did, however, provide some degree of differentiation between different brick dusts that was consistent with the results of the other tests. Because Brick Dust A formed less solid matter, it presumably formed less calcium silicate hydrate and was, as a result, less pozzolanic.

It is particularly important to use a control in this test, as most of the determinations are made through visual observations. Using a non-pozzolanic control allows one to detect differences in material consistency, such the flocculent nature of the calcium silicate hydrate, and to note different rates of settlement between a non-pozzolanic material and a pozzolan. It is also important to monitor the specimens for seven days or more to allow enough time for the pozzolanic reaction to occur. Formation of reaction products was not immediately apparent in Brick Dust A, but an increase in solid matter, although subtle, was observed at the end of the testing period.

This test is different from the others because it does not relate to any specific property that brick dust imparts to mortar. The results are not particularly useful for anything other than a simple determination of whether the material will react. The results of this test do not necessarily offer an indication as to how a pozzolan will affect a mortar's strength, setting rate, or hydraulic properties. More testing with a number of different pozzolanic materials would be necessary to

make correlations between these properties and the ability of a material to react with lime visually. However, for this particular test, degree of reactivity as indicated by formation of calcium silicate hydrates was directly proportional to setting time and compressive strength.

8.5 LIME-POZZOLAN STRENGTH DEVELOPMENT

The most commonly used and accepted method of testing pozzolanicity is by measuring the strength that a pozzolan imparts to a mortar. Strength enhancement is derived from the cementing calcium silicate hydrates formed in the reaction between pozzolan and lime. More reactive pozzolans result in higher strength mortars. Performance-based mechanical testing is often used because it is practical and the results are more easily interpreted than quantitative chemical testing.

Because strength is a property that is easily measured, strength testing yields very specific data (compressive strength = maximum load/surface area of test specimen) that can be compared to a non-pozzolanic control and can also differentiate between pozzolans to determine degrees of pozzolanicity. The distinction between the pozzolanic activity of Formulation A and B was made very clear in this test. Formulation B tested 8.3 times stronger in compression than Formulation A, indicating that the Belden brick dust is a significantly more reactive pozzolan than the Williamsburg brick dust. These results are consistent with the results of the other three pozzolanicity tests performed for this testing

program, but the compressive strength test makes the huge distinction between the pozzolanicity of the two materials (at 30 days of curing) very apparent.

ASTM C 593 *Standard Specification for Fly Ash and Other Pozzolans for Use with Lime* gives a compressive strength requirement of 600 psi at 28 days for pozzolans to be used with lime. However, the standard also requires very specific curing conditions at high temperatures that were not possible for this testing program. Also, the strength requirement of the ASTM may not be relevant for conservation mortars that often do not necessarily need to reach high compressive strengths. If the samples were to be judged based on ASTM 593, Formulation B would pass at 756 psi and Formulation A would fail at 113 psi..

While Formulation A had a relatively low compressive strength, this does not necessarily indicate that it is not pozzolanic or is a poor pozzolan. In fact, because of the results of other tests, it is clear that Brick Dust A is pozzolanic; it would not set under water if it was not. The speed of the pozzolanic reaction is dependent upon a number of factors including the mineralogy of the pozzolan as well as the firing temperature. Pozzolanic reactions can be very slow to occur, especially for brick dusts (e.g. Williamsburg brick dust) fired at higher temperatures. It is likely that, if allowed to cure for a longer period before testing, the pozzolanic reaction would eventually result in a substantial increase in compressive strength. It is recommended that compressive strength testing be performed on the mortar cubes again in at least 90 days.

Overall, the lime-pozzolan strength development test was successful in determining and quantifying pozzolanicity. When performing this test at 30 days,

however, it must be remembered that this may not be a sufficient amount of time for a pozzolanic reaction to develop to its highest potential, and the test could give low compressive strength results that may not necessarily be indicative of reactivity. Brick dusts that do not yield extremely high compressive strength results after 30 days should not be labeled non-pozzolanic or eliminated unless high early strength development is a necessary property for a given project for which the brick dust is being tested. In conservation work, high compressive strength is not always a necessary property, and other properties such as setting time and ability to set without carbon dioxide may be more important.

8.6 SYNTHESIS

The results of all tests indicated that Brick Dust B (Belden) was more pozzolanic based on its effects on the tested properties. The degree of sensitivity varied among different tests, but each test was able to confirm pozzolanicity either through the absence or presence of a particular reaction (i.e. set under water and lime combination) or comparison with a control (i.e. set time and compressive strength). The difference in pozzolanicity of Brick Dust A and Brick Dust B is probably due to the difference between the firing temperatures of the two bricks (Brick Dust B was fired about 130°C lower than Brick Dust A). The difference in pozzolanicity could also be affected by the chemical and mineralogical differences between the two.

8.7 STATISTICAL ANALYSIS

Statistical analysis is important in experimental programs to determine the reliability of data. Two forms of statistical analysis were employed in this study: standard deviation and the f-test. Standard deviation represents the typical distance from any point in a single set of data to the average of that data set. For the setting time test, set under water test, and lime combination test, standard deviation was calculated for each data reading of each formulation. Because the lime combination test was based on qualitative visual observation and had no true or variable unit data measurements, it was not subjected to statistical analysis.

The standard deviation calculations for the setting time and set under water tests show that the data is within a reasonable range, with no more than 10 standard deviations for any reading. This indicates that the measurements were reliable and consistent with little variation from the mean. The standard deviation for the lime-pozzolan strength development tests showed a greater standard deviation. Formulation A had a standard deviation of 35.51 when tested in compression (measured in psi) at 30 days. Formulation B had a standard deviation of 182.83. Formulation C had a standard deviation of 29.98. Wide ranges of compressive strength measurements can be explained by imperfections in the test specimens themselves. Imperfect molding and the creation of striations or air pockets in the packing method of the molds could lead to vulnerable areas within the structure of the samples, causing points of failure

upon compressive loading. The high standard deviation of Formulation B reflects the early failure of Sample B2 when compared to the others; its compressive strength was found to be only about half of the others. Standard deviation calculations for each test are found in their respective appendices.

The F-test is another type of statistical analysis that is used to determine whether two different data sets have significantly different variances. The F-test yields a two-tailed probability that the variances of two different data sets are not significantly different. The value is a comparison of the variances from the two different data sets, and the closer the value is to 1, the less significantly different the variances are. This test was used to compare the data from the different formulations for compressive strength and set time tests. Set under water was not subjected to the F-test because of the absence of variation in setting time for Formulation B and the lack of data for Formulation C because it did not set. The lime combination test, again, was not subjected to statistical analysis because of lack of quantitative data. The following F values were calculated using data from these tests:

Table 8.2 F-Test Results

Compared Groups	Compressive Strength	Set Time
A/B	0.01	0.86
A/C	0.76	0.04
B/C	0.00	0.03

The low F-test values indicate that the variances of the data sets are significantly different. The only groups that were not significantly different were Formulation A/

Formulation C in Compressive Strength and Formulation A/ Formulation B in set time. In compressive strength, the low values can be attributed to the low value due to the early failure of sample B2, explained above. In Set time, the low value is owing to the control sample rather than the pozzolanic samples. Because Formulation C was so slow to set, set time was measured every 5 hours at the end of the testing period rather than every 1 or 2, as Formulation A and B were. This led to a higher variation in final set time and a large variance compared to the other formulations.

CHAPTER 9 CONCLUSIONS AND RECOMMENDATIONS

9.1 SUMMARY OF RESEARCH

The purpose of this study was to determine if brick dust-enhanced lime mortars typically used in architectural conservation could be practically evaluated using field determinations for pozzolanicity. The testing program yielded valuable insights into the efficacy of existing testing methods for measuring the pozzolanicity of brick dust. The goal of the testing program was to evaluate the least complex of the existing tests reported in technical literature to determine their capacity to easily predict the ability of a given brick dust to produce a pozzolanic reaction when combined with lime. The following conclusions can be made regarding the efficacy of the pozzolanicity tests explored in this research:

1. The time of setting test is an effective method of measuring pozzolanicity. It allows a positive/negative determination of pozzolanicity when compared to a non-pozzolanic control, but is also sensitive enough to differentiate between pozzolanic materials in order to assign relative degrees of pozzolanicity. It meets most of the criteria for a practical field test, including low cost, low complexity, low technical proficiency, little time, and low equipment requirement.
2. The underwater set test is an effective method of determining pozzolanicity. While it allows a simple positive/negative determination with great accuracy, it is not sensitive enough determine differences among

pozzolans to rank them according to degree of pozzolanicity. It does meet all requirements for a practical field test including low cost, low complexity, low technical proficiency, little time, no controlled environment, and low equipment requirement.

3. The lime combination test is an effective method of determining pozzolanicity quickly in the field, but it does not yield reliably quantifiable results and is not very conclusive for pozzolans that are not highly reactive and exhibit more subtle reactions. It does allow some differentiation between pozzolans based on level of reactivity. It meets all criteria for a practical field test including low cost, low complexity, low technical proficiency, little time, no controlled environment, and low equipment requirement.
4. The lime-pozzolan strength development test is an effective method of determining pozzolanicity and differentiating between brick dusts based on strength enhancement. It may not be conclusive at only 30 days, however, for all varieties of brick dust, as the pozzolanic reaction is slow to occur in some brick dusts depending on mineralogy and firing temperature. Compressive strength testing meets the following criteria for a practical field test: low complexity and rapid results.

Pozzolanicity of brick dust is a complex function of firing temperature, mineralogy, chemical composition, and particle size. Particle size is easily determined and controlled, but firing temperature and mineralogy of recycled

bricks is difficult, if not impossible, to determine. The pozzolanicity tests evaluated in this research were all successful to some extent in determining the pozzolanicity of brick dust, especially in situations in which the clay mineralogy and firing temperature of the brick is unknown. Any of these tests could be utilized for field testing alone or in combination, but the selection of tests for determination of pozzolanicity will ultimately depend on the requirements of a particular project in terms of the necessary degree of precision and quantifiable results and the desired properties of the pozzolanic mortar.

When evaluating brick dust for its suitability to act as a pozzolan in conservation mortars, it is critical to perform a series of salt tests to determine the presence of nitrates, sulfates, and chlorides that may be present in the brick. Salt tests should be a complement to any pozzolanicity testing program, and no brick dust determined to have excessive salts should be mixed into a mortar. Introduction of salts into a masonry system via the mortar will cause salt crystallization and resulting mechanical damage to masonry as well as efflorescence on the face of stone or brick walls. Testing for salts is a simple process that can be done in the field with the use of semi-quantitative commercial salt test strips. The strips are simply saturated with a solution of brick dust and deionized water and determinations of relative quantity of salts are made immediately by observing color change on the strip similar to pH strip testing.

9.2 RECOMMENDATIONS FOR FUTURE WORK

Because of time constraints, this study did not explore the issue of pozzolanicity testing of brick dust to its fullest extent. There are still many opportunities for further investigation that could be accomplished in the future. The recommendations for future work were divided into two categories: characterization and testing.

9.2.1 CHARACTERIZATION

It was the original intent of this study to provide a precise chemical analysis of the cured pozzolanic mortars in order to identify the type and quantity of reaction product resulting from pozzolanic reactions of Formulation A and Formulation B. This would have provided the opportunity to identify exactly what compounds were being formed and how they affected the properties of the mortars. Identifying the reaction products and their relative quantities would allow one to more accurately assess the pozzolanicity tests by correlating results with known hydraulic compounds.

It was not possible to perform this analysis because of time constraints. It would be very beneficial, however, to perform material analysis of the pozzolanic mortars in the future after they have cured for a year or more, and to correlate these results with the results of the testing program. The best method for this determination is differential thermal analysis (DTA). DTA was briefly discussed as a testing method in Chapter 4 *Evaluation and Testing of Pozzolans*. By

heating the crushed composite sample at a constant known temperature, in comparison with an inert reference material, DTA results in a thermogram that displays exotherms and endotherms (peaks and valleys) that correspond to the phase changes in the material at different temperatures. These characteristic patterns provide a “fingerprint” for identifying the reaction products.[101]

Another recommendation for characterization of cured pozzolanic mortars is through the use of optical microscopy. Through scanning electron microscopy or examination of thin sections, hydraulic reaction products could be visually identified and their relative quantities estimated by observing microstructure and new crystal growth as well as the “reaction layers” noted in the work of Baronio and Binda.[102] This type of characterization is most promising after a longer period of curing when the reaction products have had the opportunity to fully form.

9.2.2 FUTURE TESTING

Another test for lime combination that was not incorporated into this study because of time constraints can be recommended for future study. This test could potentially correlate the reactions observed in this testing program with the brick dusts’ ability to fix lime, but in a more quantitative and standardized manner

[101] V.S. Ramachandran, Ralph M. Paroli, James J. Beaudoin, and Ana H. Delgado, *Handbook of Thermal Analysis of Construction Materials*, Norwich, NY: Noyes Publications, 2002, pp. 323-327.
[102] G. Binda, L. Baronio, and N. Lombardini, *The Role of Brick Pebbles and Dust in Conglomerates Based on Hydrated Lime and Crushed Brick*, Seventh North American Masonry Conference, University of Notre Dame, Indiana, 1996.

than the lime combination test that was selected for this testing program. A standardized method for assessing lime combination is the Chapelle method described in Chapter 4 *Evaluation and Testing of Pozzolans*. The test procedure involves placing the pozzolan in a saturated solution of calcium hydroxide (limewater) and then measuring the decrease in saturation of the solution through titration. A highly unsaturated solution at the end of the testing period indicates that calcium hydroxide has been fixed by pozzolan. Rather than measuring saturation of the solution through complicated titration procedures, it is recommended that commercial calcium test strips be used to measure the amount of calcium in the solution. Instructions provided with commercial calcium test strips should be followed for measuring calcium content, and the limewater solution may need to be diluted prior to testing depending on the parameters of the test strips. Assuming that the test strips have adequate sensitivity to distinguish between different saturation levels of the two brick dust solutions, this test could help explain the differences in the pozzolanicities of the two brick dusts found in this testing program based on their ability to combine with lime.

An extension of this study could also include further testing of cured pozzolanic mortars to determine long-term effects of the brick dusts on the properties of lime mortars and correlate them to the results obtained in the pozzolanicity tests. This would be very pertinent to the research, as it is known that the pozzolanic reaction can sometimes be slow to develop. This would have the most significant implications for strength development, as the ultimate strength obtained by a pozzolanic mortar is undoubtedly higher than it is at 30

days. Furthermore, it has been suggested that bricks fired at the higher temperature range for pozzolanicity (around 950° C) can develop higher strengths over time although their initial strength may not be as high.[103] If strength testing could be repeated at a later date, it may indicate that Formulation A would develop higher strength after a longer curing period, as the Williamsburg brick is known to have been fired around 950° C. It is recommended that another phase of compressive strength testing be performed in no less than 180 days and, preferably, samples be allowed to continue curing in humid conditions, as the pozzolanic reaction is known to develop to its greatest potential when allowed a long, humid cure.[104]

For a more comprehensive evaluation of the tests selected for this testing program, more variables could be introduced in a second phase of testing. Formulations with different variables could allow a more definite determination of the tests' degrees of sensitivity. For example, performing the pozzolanicity tests on samples with varying amounts of brick dust in the mix could determine whether a given test is adequate in detecting the differences in proportions, as the Smeaton project demonstrated that higher proportions of brick dust create

[103] D.B Hughes and D.C. Sugden, "The Use of Brick Dust as a Pozzolanic Additive to Hydraulic Lime Mortars," *Historic Mortars: Characterization and Tests,* Proceedings of the International RILEM Workshop, 2000, pp. 351-367.
[104] A. Elena Charola, Paulina Faria Rodrigues, Andrew R.McGhie and Fernando M.A.Henriques, *Pozzolanic Components in Lime Mortars: Correlating Behaviour, Composition and Microstructure*, 6th International Symposium on the Conservation of Monuments in the Mediterranean Basin, Lisbon, 2004.

more pronounced effects in the properties of mortars.[105] If more phases of this study are to be undertaken, tests should employ both a non-pozzolanic control and a highly pozzolanic control, such as pure metakaolin produced specifically for construction purposes. This could establish an upper limit for pozzolanicity in terms of reactivity and effect on strength and setting time for comparison and would allow more sound judgments on the efficacy of the tests.

[105] Jeanne Marie Teutonico, Iain McCaig, Colin Burns, John Ashurst, "The Smeaton Project: Factors Affecting the Properties of Lime-based Mortars," *APT Bulletin*, Vol. 25, No. 3/4. (1993), pp. 32-49.

BIBLIOGRAPHY

"About Natural Hydraulic Limes." St. Astier Natural Hydraulic Lime website: http://www.limes.us/products.php, accessed April 1, 2011.

Ashall, G., R. N. Butlin, J. M. Teutonico, and W. Martin. *Development of Lime Mortar Formulations for Use in Historic Buildings* (Smeaton Project). Proceedings of the Seventh International Conference on Durability of Building Materials and Components. Stockholm, 1996, pp. 353-360.

Ashurst, John. *Mortars, Plasters and Renders in Conservation.* 2nd ed. Ecclesiastical Architects' and Surveyors' Association, 2002.

Badogiannis, E., G. Kakali, and S. Tsivilis. "Metakaolin as Supplementary Cementitious Material, Optimization of Kaolin to Metakaolin Conversion." *Journal of Thermal Analysis and Calorimetry,* Vol. 81, 2005, pp. 457-462.

Baronio, Luigia and Guilia Binda. "Byzantine Concretes: The Role of Thick Mortar Joints Containing Crushed Brick." *Concrete: From Material to Structure.* Proceedings of the RILEM International Conference, Arles, France, 1996, pp. 442-460.

Baronio, Guilia and Luigia Binda. "Characterization of Mortars and Plasters of Ancient Monuments of Milan." *The Masonry Journal,* Vol. 7 No. 1, 1988, pp. 48-54.

Baronio, Guilia and Luiga Binda. "Study of the Pozzolanicity of Some Bricks and Clays." *Construction and Building Materials,* Vol. 11 No. 1, 1997, pp. 41-46.

Binda, G., L. Baronio, and N. Lombardini. *The Role of Brick Pebbles and Dust in Conglomerates Based on Hydrated Lime and Crushed Brick*. Seventh North American Masonry Conference. University of Notre Dame, Indiana, 1996.

Baronio, G., L. Binda, C. Tedeschi. *Microscopy Study of Byzantine Mortars: Observation of Reaction Layers Between Lime and Brick Dust*. Seventh Euroseminar on Microscopy Applied to Building Materials, Milan, 1999.

Boffey, Geoffrey and Elizabeth Hirst. "The Use of Pozzolans in Lime Mortar*."* *Journal of Architectural Conservation*, Vol. 5, No. 3, 1999, pp. 34-42.

"Brickmakers," Colonial Williamsburg website, online at http://www.history.org/almanack/life/trades/tradebri.cfm, accessed March 23, 2011.

Bugini, R. and A. Salvatori. "Investigation of the Characteristics and Properties of Cocciopesto from the Ancient Roman Period." *Conservation of Stone and Other Materials.* Proceedings of the International RILEM/UNESCO Conference, Paris, 1993, pp. 386-393.

Charola, A. Elena, Paulina Faria Rodrigues, Andrew R.McGhie and Fernando M.A.Henriques. *Pozzolanic Components in Lime Mortars: Correlating Behaviour, Composition and Microstructure*. 6th International Symposium on the Conservation of Monuments in the Mediterranean Basin, Lisbon, 2004.

Cowan, Henry J. *The Science and Technology of Building Materials.* NY: Van Nostrand Reihnold Company, 1988.

Cowper, A. D. *Lime and Lime Mortars.* Shaftsbury, UK: Donhead Publishing Co., 1927.

Davis, Raymond E. *A Review of Pozzolanic Materials and their Use in Concrete.* Symposium on Use of Pozzolanic Materials in Mortars and Concretes. ASTM Special Technical Publication No. 99, 1950.

Day, Robert. *Pozzolans for Use in Low Income Housing*: *A State of the Art Report Prepared for the International Development Research Centre*. Ottawa, Canada, 1990.

Eckel, Edwin Clarence. *Cements, Limes, and Plasters: Their Materials, Manufacture, and Properties*, 2nd ed. NY: John Wiley and Sons, 1922.

Elert, Kerstin, Carlos Rodriguez-Navarro, Eduardo Sebastian Pardo, Eric Hansen, and Olga Cazalla. "Lime Mortars for the Conservation of Historic Buildings." *Studies in Conservation*, Vol. 47, No. 1. (2002), pp. 62-75.

Gibbons, Pat. "Pozzolans for Lime Mortars." *The Building Conservation Directory*, 1997, online at www.buildingconservation.com. Accessed February 27, 2011.

Gibbons, Pat. *Technical Advice Note*: *Preparation and Use of Lime Mortars.* Revised edition 2003. The Scottish Lime Centre. Edinburgh, Scotland: Crown Copyright, 2003.

Griffin, Isobel. *Grouts for the Conservation of Architectural Surfaces, Literature Review*. Prepared for the Getty Conservation Institute, May 2005.

Griffin, Isobel. "Pozzolanas as Additives for Grouts: An Investigation of their Working Properties and Performance Characteristics." *Studies in Conservation,* Vol. 49 No. 1, 2004, pp. 23-34.

He, Changling, Bjarne Osbeack, Emil Macovicky. *"*Pozzolanic Reactions of Six Principle Clay Minerals: Activation, Reactivity Measures, and Technological Effects." *Cement and Concrete Research*, Vol. 25, No. 8, 1995, pp. 1691-1702.

Hughes, D. B. and D.C. Sugden. "The Use of Brick Dust as a Pozzolanic Additive to Hydraulic Lime Mortars." *Historic Mortars: Characterization and Tests,* Proceedings of the International RILEM Workshop, 2000, pp. 351-367.

"Hydraulicity and Properties of St. Astier Natural Hydraulic Lime." 2006. St. Astier Natural Hydraulic Lime website: http://www.stastier.co.uk/nhl/info/pdfs/Hydraulicity_and_Properties_of_NHL.pdf, accessed April 1, 2011.

Lea, F. M. *The Chemistry of Cement and Concrete*, 3rd edition. NY: Chemical Publishing Company, 1971.

Livezy, Paul. "Hydraulicity." *The Building Conservation Directory*. Online at http://www.buildingconservation.com/articles/hydraulicity/hydraulicity.htm. 2003. Accessed March 31, 2011.

McCarter, W. J. and D. Tran. "Monitoring Pozzolanic Activity by Direct Activation with Calcium Hydroxide.*" Construction and Building Materials,* Vol. 10 No. 3, 1996, pp. 179-184.

Moran, W. T. and J. L. Gilliand. *Summary of Methods for Determining Pozzolanic Activity.* Symposium on Use of Pozzolanic Materials in Mortars and Concretes. ASTM Special Technical Publication No. 99, 1950.

Murray, H.H. *Applied Clay Mineralogy: Occurrences, Processing, and Application of Kaolins, Bentonites, Palygorskite-sepiolite, and Common Clays*. Amsterdam: Elsevier publications, 2007.

Pinheiro, Igor S., Luiz C. Montenegro, and Adriana G. Gumieri. *Pozzolanic Activity of Red Recycled Bricks.* Second International Conference of Sustainable Construction Materials and Technologies, Ancona, Italy, June, 2010.

Ramachandran, V. S., Ralph M. Paroli, James J. Beaudoin, and Ana H. Delgado. *Handbook of Thermal Analysis of Construction Materials*, Norwich, NY: Noyes Publications, 2002.

Searle, Alfred B. *Modern Brickmaking*. London: Scott, Greenwood & Son, 1911.

Stark, Jochen and Katrin Bollmann. "Delayed Ettrignite Formation in Concrete," (paper) Bauhaus University, Germany.

Technical Brief: *Testing Methods for Pozzolans.* Practical Action: Schumacher Center for Technology and Development, online at www.practicalaction.org.

Teutonico, Jeanne Marie, Iain McCaig, Colin Burns, and John Ashurst. "The Smeaton Project: Factors Affecting the Properties of Lime-based Mortars." *APT Bulletin*, Vol. 25, No. 3/4. (1993), pp. 32-49.

Torraca, Georgio. *Lectures on Material Science for Architectural Conservation*. Los Angeles: The Getty Conservation Institute, 2009.

Vicat, L. J. *Mortars and Cements.* Shaftsbury, UK: Donhead Publishing Co., 1997. (originally published 1887).

Vitruvius, Marcus. *Ten Books on Architecture*, Book II, translated by Morris Hickey Morgan. New York: Dover Publications Inc, 1960.

REFERENCED STANDARDS

American Society for the Testing of Materials. "C109 Standard Test Method for Compressive Strength of Hydraulic Cement Mortars.*" Annual Book of ASTM Standards*. West Conshohocken, PA: American Society for the Testing of Materials, 2000.

American Society for Testing and Materials. "C 141-97 Standard Specification for Hydraulic Hydrated Lime for Structural Purposes." *Annual Book of ASTM Standards.* Vol .04.01. West Conshohocken, PA, ASTM 1997.

American Society for Testing of Materials. “C191-99 Standard Test Method for Time Setting of Hydraulic Cement by Vicat Needle.” *Annual Book of ASTM Standards*. Vol. 04.01. West Conshohocken, PA, ASTM 2000.

American Society for Testing and Materials. “C 219-01 Standard Terminology Related to Hydraulic Cement.” *Annual Book of ASTM Standards.* Vol .04.01. West Conshohocken, PA, ASTM 2000.

American Society for Testing and Materials. “C311 - 07 Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete.” *Annual Book of ASTM Standards.* Vol.04-02. West Conshohocken, PA, ASTM 2007.

American Society for Testing and Materials. “C446 Standard Test Method for Effectiveness of Pozzolans or Ground Blast-Furnace Slag in Preventing Excessive Expansion of Concrete Due to the Alkali-Silica Reaction.” *Annual Book of ASTM Standards.* Vol.04-02. West Conshohocken, PA, ASTM 2008.

American Society for Testing and Materials. “C 593-95 Standard Specification for Fly Ash and Other Pozzolans for Use with Lime.” *Annual Book of ASTM Standards.* Vol. 04.01. West Conshohocken, PA, ASTM 2000.

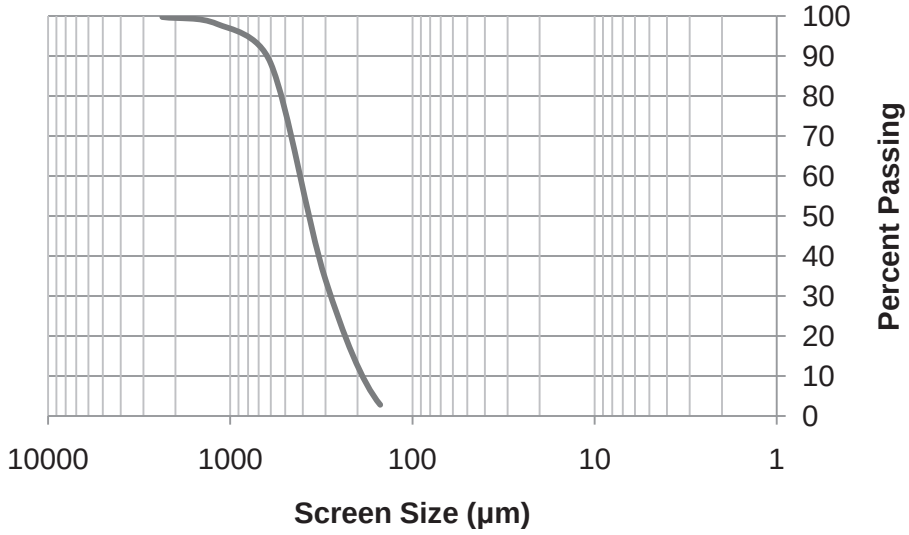
American Society for Testing and Materials. “C618 - 08a Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete.” *Annual Book of ASTM Standards.* Vol.04-02. West Conshohocken, PA, ASTM 2008.

APPENDIX A: TESTING AND SAMPLE SCHEDULE

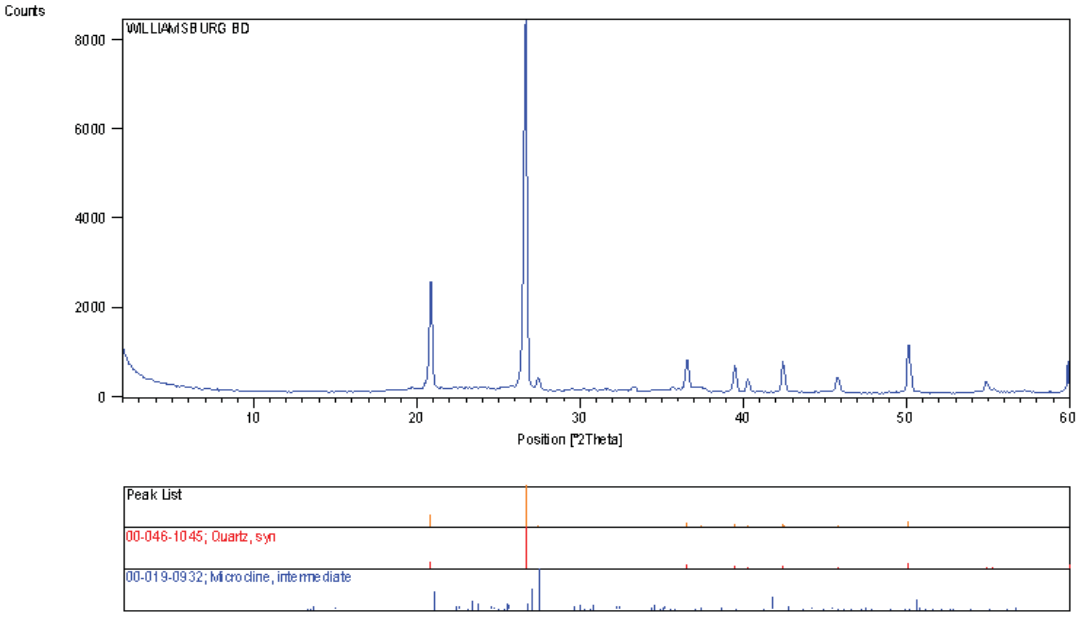
Test	Property Measured	Standard	Referenced Standards	# of Samples	Size of samples	Molds	Proportions	Equipment	Materials	Start Date	Run Time	Description
X-Ray Diffraction	chemical composition	n/a	n/a	2 (1 for each brick dust)	minute quantity	n/a	n/a	n/a	minute quantities of each brick dust ground to pass 400 um sieve	4-19-11	1 day	X-ray diffraction performed at LRSM to determine mineralolgy of brick dusts
Consistency	flow	ASTM C 593	ASTM C 109, C 230	6 (2 for each mortar formulation)	4" base diameter, 2 3/4" top diamter, 2" depth	truncated cone	1:3:1 lime: sand: BD	flow table	Use mortar from compressive strength test	3-19-11	1 minute	Use flow table to determine consistency of mortar before molding for compressive strength and set time test
Compressive Strength	compressive strength	ASTM 593 and ASTM 109	C 230, C 305, C349, C 511, C 670, C 778, C 1005	45 (15 Form. A, and 15 Form. B, 15 Form. C)	2 in cube (50 mm)	2 inch wooden mold	1:3:1 lime: sand: BD	Instron Testing Machine	hydrated lime, BD A, BD B, marble dust, sand	4-18-11	28 days to set, 1 day for testing	Test compressive strength of samples after 28 days and compare to control to determine pozzolan strength enhancement
Set Time Test	set time	ASTM 191	C 191, C 305, C 266, C 490, C 187	9 (3 Form. A, 3 Form. B, 3 Form. C)	70 mm/60 mm by 40 mm	conical ring with glass plate	1:3:1 lime: sand: BD	Vicat apparatus	hydrated lime, sand, BD A, BD B marble dust	3-19-11	less than 1 week	Measure initial and final set using Vicat apparatus
Underwater set test	set under water	Feret method (1925) and ASTM C 191	n/a	10 (3 Form. A, 3 Form. B, 3 Form. C)	volume of glass jar	cylindrical glass jars	1:4 lime: BD paste	Vicat apparatus	hydrated lime, BD 1, BD 2, marble dust, mineral oil, limewater	3-30-11	less than 1 week	Measure initial and final set time under water using Vicat apparatus
Lime Absorption Test	Ability to fix lime	None; Cowpers 1927	n/a	15 (5 Form. A, 5 Form. B, 5 Form. C)	minute quantity	none	n/a	15 50 mL graduated cylinders with stoppers	2.5 g each of BD A, BD B, and marble dust, 4.5 g lime, distilled water	4/14/11	7 days	Determine volumetric increase of solid matter in cylinder every 12 hours for 7 days

APPENDIX B: PARTICLE SIZE DISTRIBUTION OF CAVA BUILDING SUPPLY BAR SAND

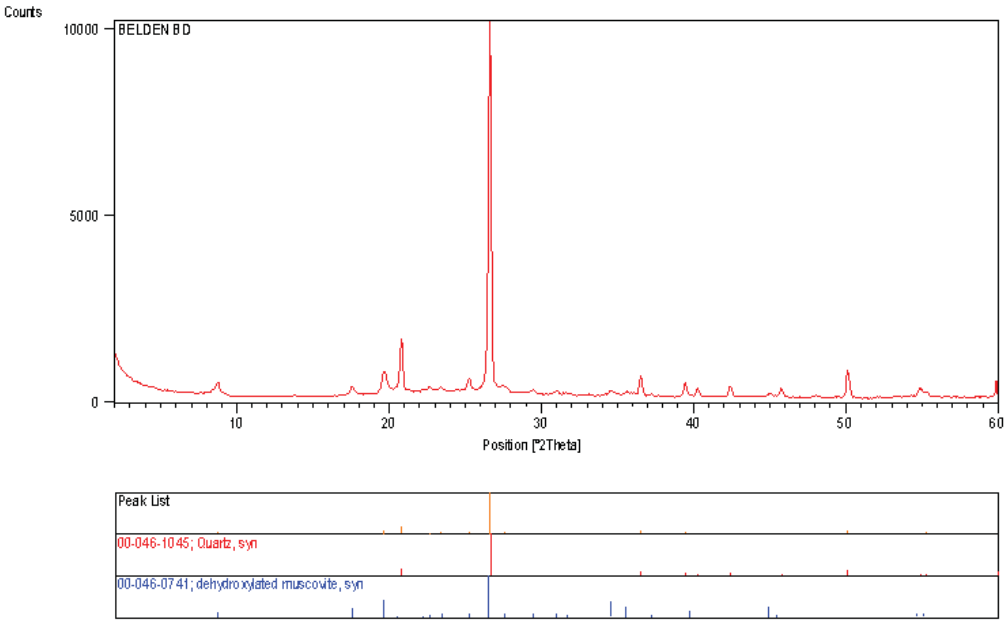
Sieve Number	Screen Size (µm)	Mass of Container (g)	Mass of sample + container (g)	Mass retained (g)	Percent mass retained	Percent on or above	Percent passing
8	2360	2.60	2.80	0.20	0.20	0.20	99.80
16	1180	2.76	4.50	1.74	1.74	1.94	98.06
30	600	2.70	12.48	9.78	9.78	11.72	88.28
50	300	2.76	56.96	54.20	54.18	65.89	34.11
100	150	2.66	34.04	31.38	31.37	97.26	2.74
200	75	2.79	4.61	1.82	1.82	99.08	0.92
Pan	0	2.76	2.78	0.02	0.02	99.10	0.90



APPENDIX C: XRD SPECTRA OF BRICK DUSTS



Williamsburg Brick Dust



Belden Brick Dust

APPENDIX D: PROPORTIONS AND QUANTITIES OF COMPONENTS FOR FLOW, SETTING TIME, AND COMPRESSIVE STRENGTH SAMPLES

PROPORTIONS BY VOLUME

Formulation	Hydrated High Calcium Lime	Bar Sand	Williamsburg brick dust	Belden brick dust	Marble dust
A	1	3	1	--	--
B	1	3	--	1	--
C	1	3	--	--	1

MEASURED QUANTITIES OF COMPONENTS

Formulation	High Calcium Lime (g)	Bar Sand (g)	Williamsburg brick dust (g)	Belden brick dust (g)	Marble dust (g)	Water (mL)
A	749.95	7149.37	1234.17	--	--	1750
B	751.56	7150.56	--	1152.23	--	1550
C	751.63	7150.56	--	--	1152.08	1360

APPENDIX E: FLOW TEST DATA

FLOW MEASUREMENTS

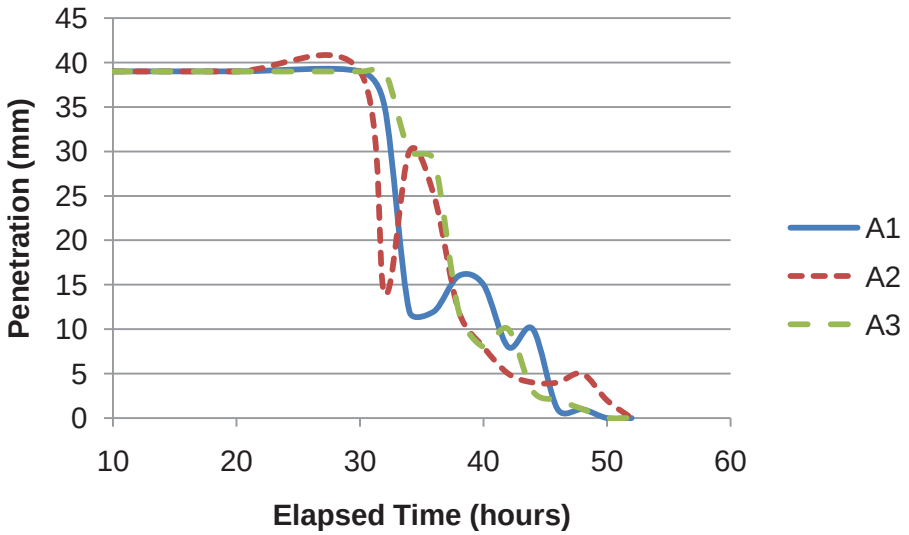
Sample	L1 (mm)	L2 (mm)	L3 (mm)	L4 (mm)	% flow	average
A1	108.38	112.83	114.45	114.26	10.7%	10.65%
A2	110.51	113.77	113.63	111.38	10.6%	
B1	109.0	107.62	108.49	107.64	6.48%	5.67%
B2	105.20	106.51	106.67	107.74	4.85%	
C1	106.14	107.47	107.79	107.55	5.55%	5.52%
C2	106.02	107.72	107.45	107.51	5.49%	

APPENDIX F: SETTING TIME DATA

PENETRATION MEASUREMENTS OF FORMULATION A (WILLIAMSBURG BRICK DUST)

Time (hr)	A1 (mm)	A2 (mm)	A3 (mm)	Average (mm)	Standard Deviation (mm)
10	39	39	39	39.00	0.00
20	39	39	39	39.00	0.00
30	39	39	39	39.00	0.00
32	35	14	39	29.33	10.96
34	12	30	30	24.00	8.49
36	12	25	29	22.00	7.26
38	16	12	12	13.33	1.89
40	15	8	8	10.33	3.30
42	8	5	10	7.67	2.05
44	10	4	3	5.67	3.09
46	1	4	2	2.33	1.25
48	1	5	1	2.33	1.89
50	0	2	0	0.67	0.94
52	0	0	0	0.00	0.00

SETTING TIME OF FORMULATION A (WILLIAMSBURG BRICK DUST)

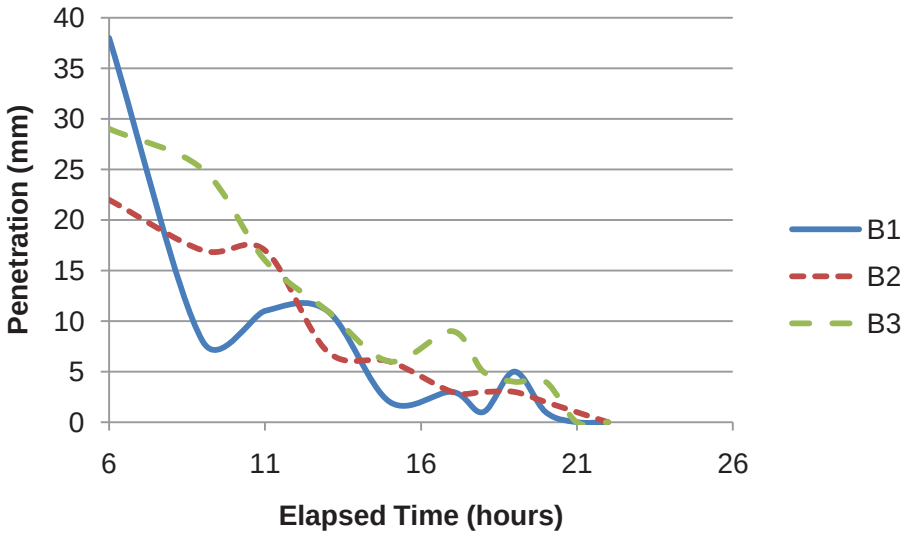


APPENDIX F: SETTING TIME DATA

PENETRATION MEASUREMENTS OF FORMULATION B (BELDEN BRICK DUST)

Time (hr)	B1 (mm)	B2 (mm)	B3 (mm)	Average (mm)	Standard Deviation (mm)
6	38	22	29	29.67	8.02
9	8	17	25	16.67	8.50
11	11	17	16	14.67	3.21
13	11	7	11	9.67	2.31
15	2	6	6	4.67	2.31
17	3	3	9	5.00	3.46
18	1	3	5	3.00	2.00
19	5	3	4	4.00	1.00
20	1	2	4	2.33	1.53
21	0	1	0	0.33	0.58
22	0	0	0	0.00	0.00

SETTING TIME OF FORMULATION B (BELDEN BRICK DUST)

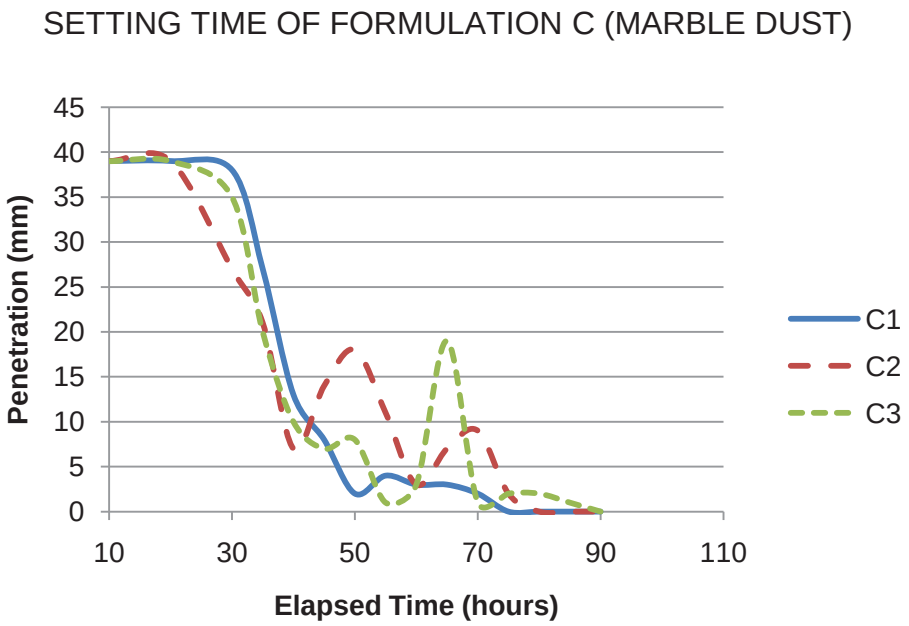


APPENDIX F: SETTING TIME DATA

PENETRATION MEASUREMENTS OF FORMULATION C (MARBLE DUST)

Time (hr)	C1 (mm)	C2 (mm)	C3 (mm)	Average (mm)	Standard Deviation (mm)
10	39	39	39	39.00	0.00
20	39	39	39	39.00	0.00
30	38	27	35	33.33	5.69
35	27	21	20	22.67	3.79
40	13	7	10	10.00	3.00
45	8	14	7	9.67	3.79
50	2	18	8	9.33	8.08
55	4	11	1	5.33	5.13
60	3	3	3	3.00	0.00
65	3	7	19	9.67	8.33
70	2	9	1	4.00	4.36
75	0	2	2	1.33	1.15
80	0	0	2	0.67	1.15
85	0	0	1	0.33	0.58
90	0	0	0	0.00	0.00

APPENDIX F: SETTING TIME DATA

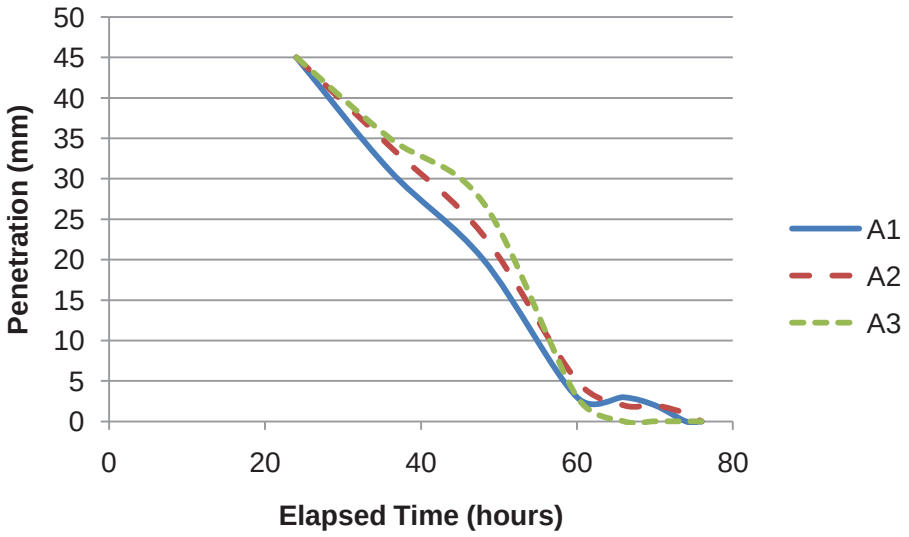


APPENDIX G: SET UNDER WATER DATA

UNDERWATER PENETRATION MEASUREMENTS OF FORMULATION A (WILLIAMSBURG BRICK DUST)

Time (hr)	A1 (mm)	A2 (mm)	A3 (mm)	Average (mm)	Standard Deviation (mm)
24	45	45	45	45.00	0.00
36	31	34	35	33.33	2.08
48	20	23	27	23.33	3.51
60	3	5	3	3.67	1.15
66	3	2	0	1.67	1.53
70	2	2	0	1.33	1.15
74	0	1	0	0.33	0.58
76	0	0	0	0.00	0.00

UNDERWATER SETTING TIME OF FORMULATION A (WILLIAMSBURG BRICK DUST)

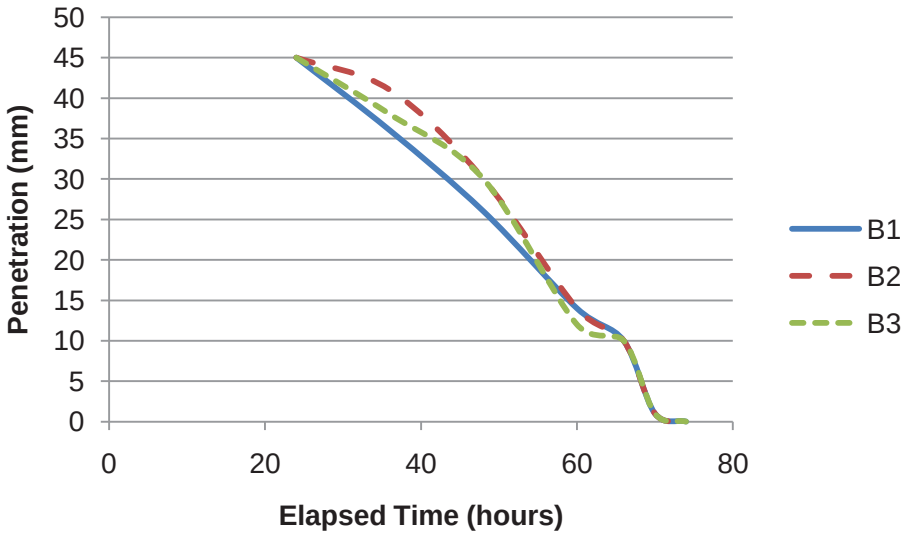


APPENDIX G: SET UNDER WATER DATA

UNDERWATER PENETRATION MEASUREMENTS OF FORMULATION B (BELDEN BRICK DUST)

Time (hr)	B1 (mm)	B2 (mm)	B3 (mm)	Average (mm)	Standard Deviation (mm)
24	45	45	45	45.00	0.00
36	36	41	38	38.33	2.52
48	26	30	30	28.67	2.31
60	14	14	12	13.33	1.15
66	10	10	10	10.00	0.00
70	1	1	1	1.00	0.00
74	0	0	0	0.00	0.00

UNDERWATER SETTING TIME OF FORMULATION B (BELDEN BRICK DUST)



APPENDIX G: SET UNDER WATER DATA

UNDERWATER PENETRATION MEASUREMENTS OF FORMULATION C (MARBLE DUST)

Time (hr)	C1 (mm)	C2 (mm)	C3 (mm)	Average (mm)	Standard Deviation (mm)
24	45	45	45	45.00	0.00
36	45	45	45	45.00	0.00
48	45	45	45	45.00	0.00
60	45	45	45	45.00	0.00
66	45	45	45	45.00	0.00
70	45	45	45	45.00	0.00
74	45	45	45	45.00	0.00
100	45	45	45	45.00	0.00
150	45	45	45	45.00	0.00
200	45	45	45	45.00	0.00

(Underwater setting time curve not included for Formulation C because there was no set.)

APPENDIX H: LIME COMBINATION TEST DATA

HEIGHT OF SOLID MATTER IN GRADUATED CYLINDER (MEASURED IN mL) FOR BRICK DUST A

		Initial Reading (mL)					2 Min After Agitation (mL)					4 Min After Agitation (mL)				
		A1	A2	A3	A4	A5	A1	A2	A3	A4	A5	A1	A2	A3	A4	A5
	AM	2.0	2.0	2.0	2.0	2.0	2.5	2.5	2.5	2.5	2.5	2.0	2.0	2.0	2.0	2.0
Day 1	PM	2.0	2.0	2.0	2.0	2.0	3.0	3.0	3.0	3.0	2.8	2.5	2.5	2.5	2.5	2.5
	AM	2.0	2.0	2.0	2.0	2.0	3.2	3.0	3.2	3.0	3.0	2.5	2.5	2.0	2.5	2.5
Day 2	PM	2.0	2.0	2.5	2.0	2.0	3.5	3.5	3.5	3.5	3.7	2.5	2.5	2.5	2.6	2.5
	AM	2.5	2.2	2.2	2.1	2.2	3.8	3.8	3.8	3.8	3.8	3.0	3.0	3.0	3.2	3.0
Day 3	PM	2.5	2.3	2.3	2.3	2.5	3.5	3.5	3.8	3.8	3.8	3.0	3.0	3.0	3.0	3.0
	AM	2.5	2.5	2.5	2.5	2.5	3.8	3.5	3.8	3.8	3.8	3.0	3.0	3.0	3.0	3.0
Day 4	PM	2.5	2.5	2.5	2.5	2.5	3.8	3.8	3.8	4.0	3.8	2.5	3.0	3.0	3.0	3.0
	AM	2.5	2.5	2.5	2.5	2.5	3.8	3.5	4.0	4.0	4.0	3.0	3.0	3.0	3.0	3.0
Day 5	PM	2.5	2.5	2.5	2.5	2.5	4.0	4.0	4.0	3.8	4.0	3.8	3.2	3.5	3.5	3.5
	AM	2.5	2.6	2.5	2.5	2.5	4.0	4.0	4.0	4.0	4.0	3.8	3.2	3.5	3.5	3.7
Day 6	PM	3.2	3.2	3.0	3.0	3.0	4.0	4.0	4.5	4.0	4.0	3.8	3.2	4.0	3.8	3.8
	AM	3.5	3.2	3.8	3.8	3.5	4.0	4.5	4.0	4.5	4.5	3.8	4.0	4.0	4.2	4.0
Day 7	PM	3.5	3.5	3.5	3.5	3.5	4.5	4.5	4.5	4.5	4.5	4.0	4.0	4.2	4.0	4.0

APPENDIX H: LIME COMBINATION TEST DATA

HEIGHT OF SOLID MATTER IN GRADUATED CYLINDER (MEASURED IN mL) FOR BRICK DUST B

		Initial Reading (mL)					2 Minutes After Agitation (mL)					4 Minutes After Agitation (mL)				
		B1	B2	B3	B4	B5	B1	B2	B3	B4	B5	B1	B2	B3	B4	B5
	AM	1.8	1.8	1.8	1.8	1.8	7.0	5.1	6.1	6.0	5.9	5.7	4.5	5.1	5.0	4.8
Day 1	PM	4.0	3.0	4.0	4.5	4.0	8.0	7.3	8.0	7.1	7.1	6.0	6.1	6.6	6.0	6.0
	AM	4.8	4.8	5.0	4.8	4.8	8.5	8.7	8.5	8.0	8.0	8.0	8.0	8.0	7.8	7.8
Day 2	PM	5.0	5.0	5.0	5.0	5.0	8.9	8.9	9.0	8.2	8.6	8.2	8.2	8.5	7.9	7.0
	AM	5.8	5.2	5.4	5.2	5.0	8.9	8.9	9.0	8.2	8.6	8.0	8.2	8.6	7.0	7.0
Day 3	PM	5.8	5.4	5.5	5.2	5.0	8.9	8.8	8.8	8.5	9.0	8.0	8.0	8.6	7.5	7.5
	AM	6.0	6.0	5.8	5.8	5.5	10.0	9.5	9.0	9.2	10.2	8.5	8.2	9.0	8.0	8.0
Day 4	PM	6.0	6.0	5.8	5.8	5.5	11.0	10.5	10.5	11.0	11.0	9.0	9.0	9.6	8.5	8.6
	AM	8.0	7.0	6.0	7.0	7.0	11.5	11.0	12.8	12.5	13.0	10.0	9.0	10.0	8.8	10.2
Day 5	PM	8.2	7.5	7.0	7.0	7.0	13.0	13.0	13.2	11.5	12.0	11.0	10.0	10.5	9.8	10.0
	AM	8.0	7.5	7.0	7.0	7.0	14.0	15.0	15.0	13.0	13.5	13.0	12.0	11.0	10.6	11.0
Day 6	PM	8.0	7.5	7.5	7.5	7.2	16.0	15.0	15.0	14.0	14.5	14.0	11.0	12.0	11.5	12.0
	AM	9.0	8.5	9.0	9.0	9.0	16.5	15.0	16.0	17.0	16.5	14.0	11.8	12.0	12.0	12.6
Day 7	PM	9.2	8.5	9.0	9.0	9.0	17.0	16.0	16.0	17.0	16.5	14.5	13.0	13.0	13.0	13.0

APPENDIX H: LIME COMBINATION TEST DATA

HEIGHT OF SOLID MATTER IN GRADUATED CYLINDER (MEASURED IN mL) CONTROL C

		Initial Reading (mL)					2 Minutes After Agitation (mL)					4 Minutes After Agitation (mL)				
		C1	C2	C3	C4	C5	C1	C2	C3	C4	C5	C1	C2	C3	C4	C5
Day 1	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.6	2.5	2.5	2.4
Day 2	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.6	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	3.0	2.5	2.5	2.5
Day 3	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	3.0	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	3.0	2.5	2.5	2.5
Day 4	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	3.0	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	3.0	3.0	2.5	2.5	2.5
Day 5	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
Day 6	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
Day 7	AM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5
	PM	1.0	1.0	1.0	1.0	1.0	n/a	n/a	n/a	n/a	n/a	2.5	2.5	2.5	2.5	2.5

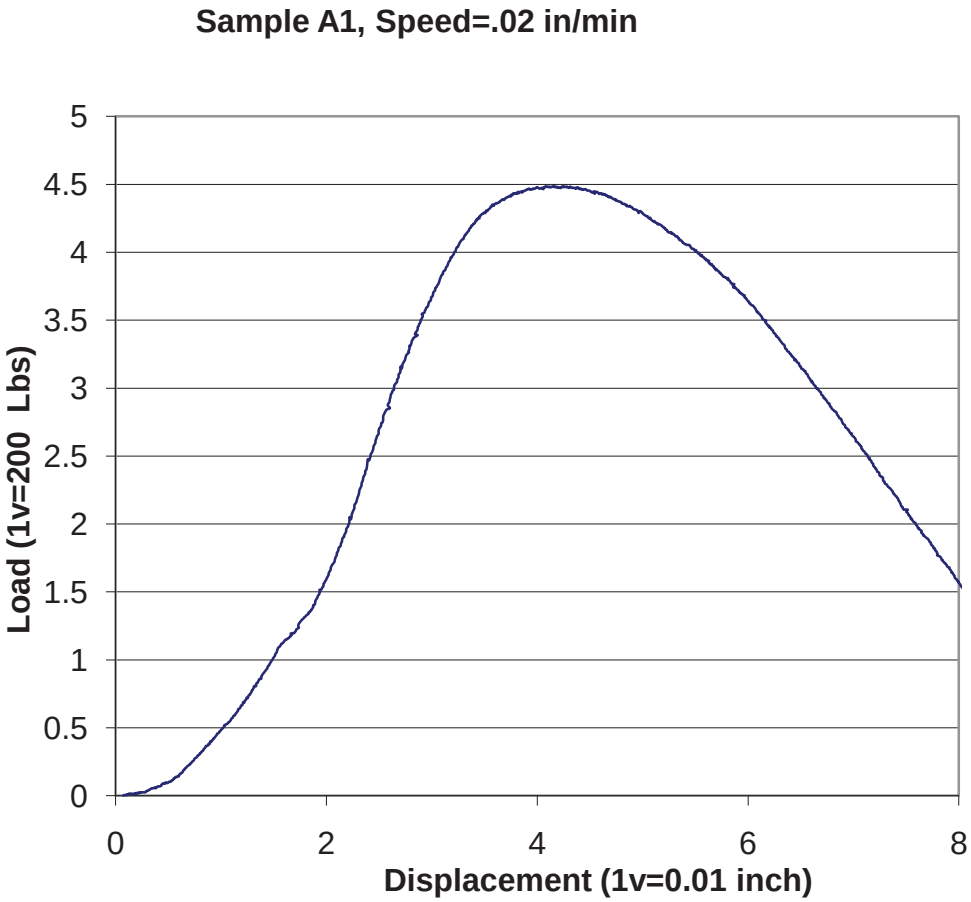
*READINGS WERE NOT TAKEN AT 2 MINUTES BECAUSE SOLID MATTER HAD NOT SETTLED ENOUGH TO MEASURE.

APPENDIX I: COMPRESSIVE STRENGTH DATA

RESULTS OF COMPRESSION TESTING

Sample	Length average (in)	Width average (in)	Surface Area (in^2)	Max load (lbs)	Comp. Strength (psi)	Standard Deviation (psi)	Average comp. strength (psi)
A1	1.99	2.01	4.01	896.48	223.57	35.31	197.56
A2	2.00	1.96	3.92	710.94	181.27		
A3	1.97	1.98	3.90	949.22	243.1		
A4	1.97	2.01	3.95	612.4	155.05		
A5	2.01	2.02	4.05	748.04	184.79		
B1	2.02	2.01	4.04	--	--	182.83	756.72
B2	2.03	2.03	4.11	2003.17	487.3		
B3	2.00	2.03	4.05	3312.99	817.22		
B4	2.01	2.00	4.02	3325.20	827.99		
B5	2.01	2.03	4.09	3654.79	894.38		
C1	2.03	2.04	4.13	260.74	63.15	29.98	112.92
C2	2.03	2.01	4.08	480.47	117.64		
C3	2.03	1.99	4.04	535.16	132.54		
C4	2.03	2.04	4.14	462.89	111.72		
C5	2.03	1.98	4.01	559.57	139.56		

APPENDIX I: COMPRESSIVE STRENGTH DATA



APPENDIX I: COMPRESSIVE STRENGTH DATA

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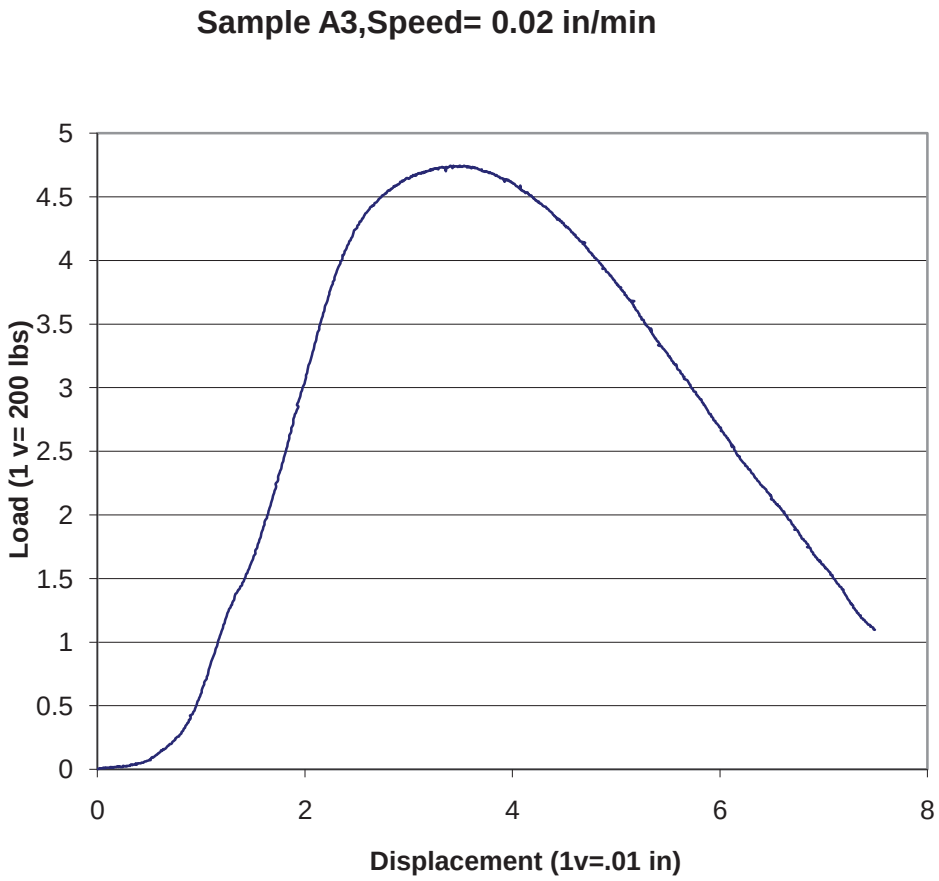
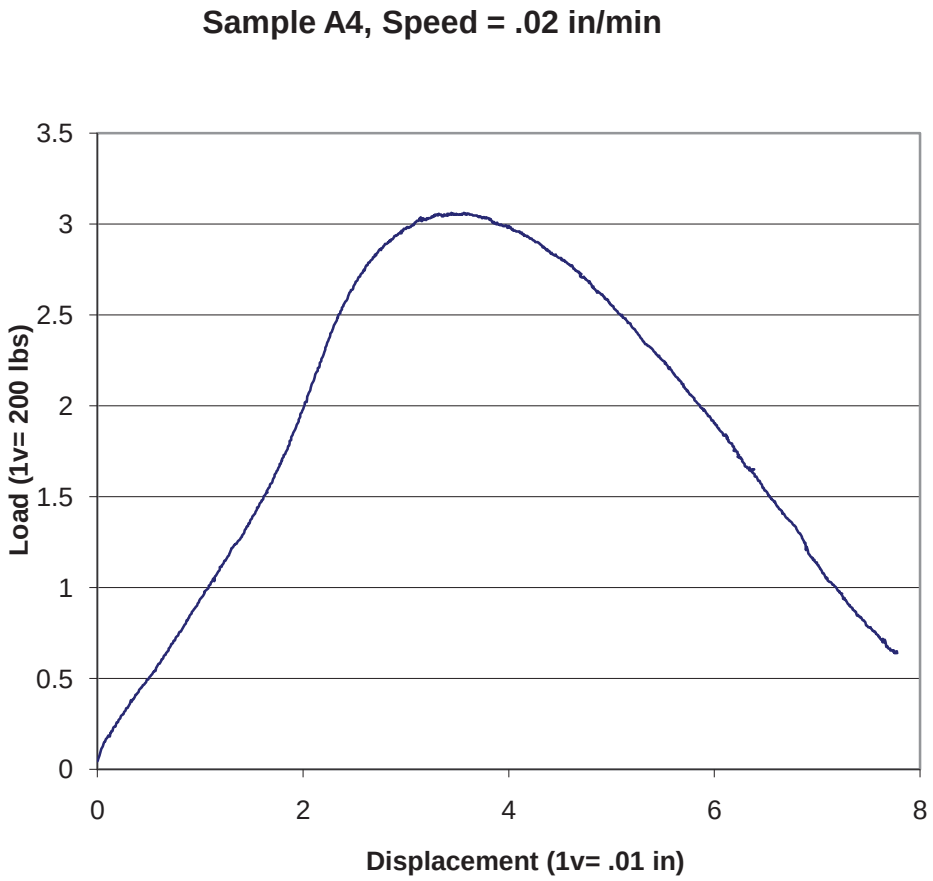
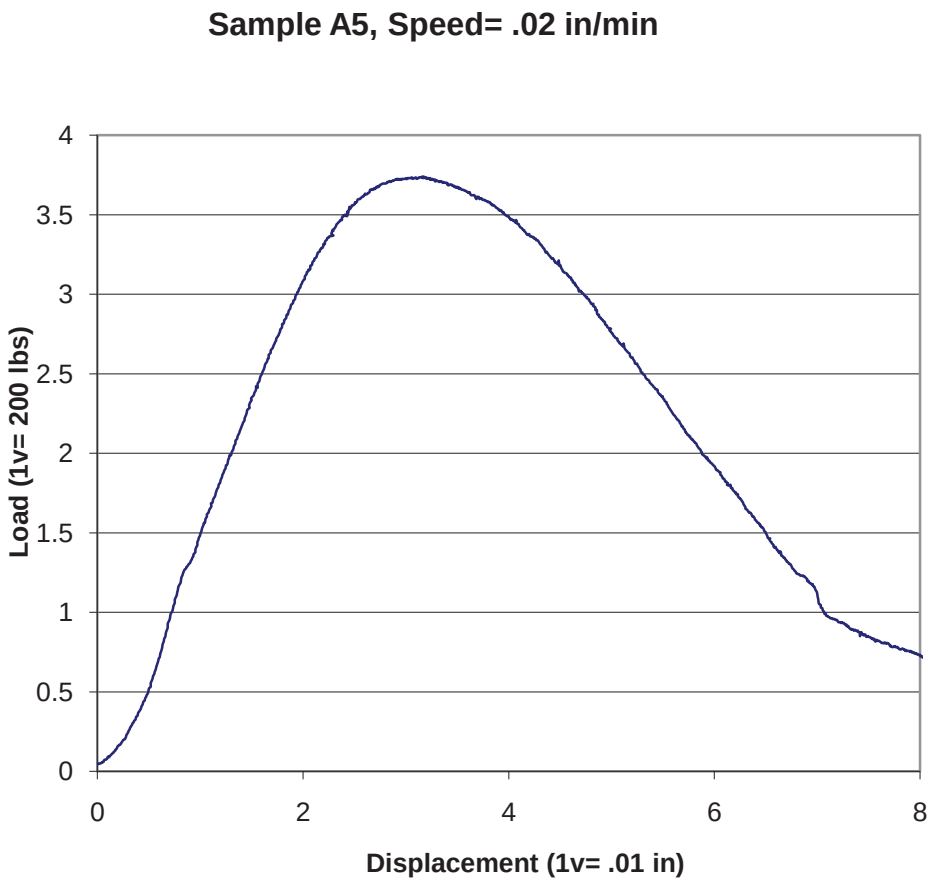


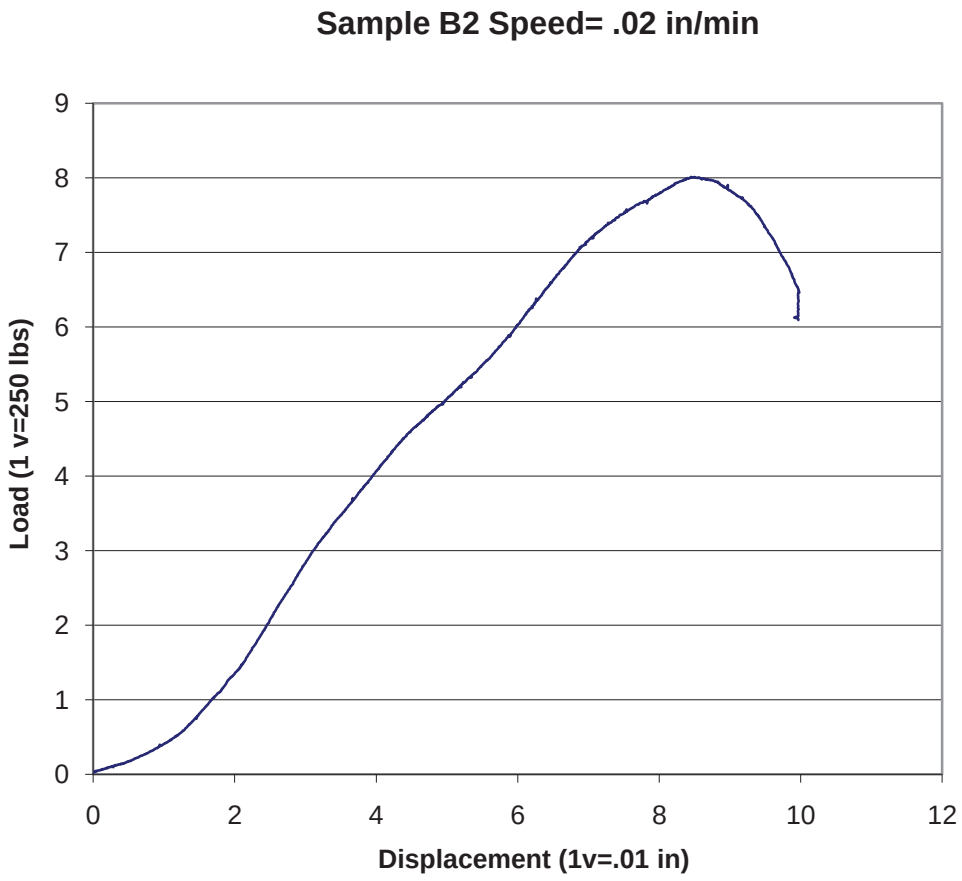
APPENDIX I: COMPRESSIVE STRENGTH DATA



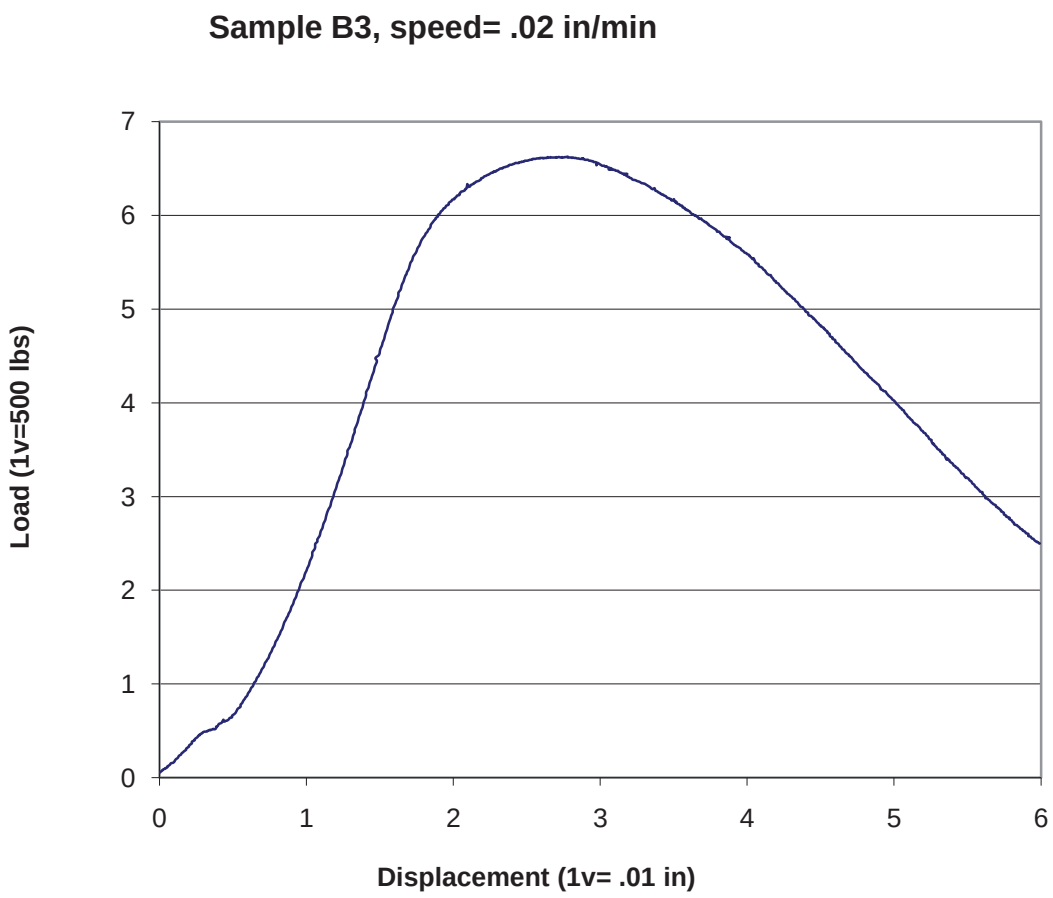
APPENDIX I: COMPRESSIVE STRENGTH DATA



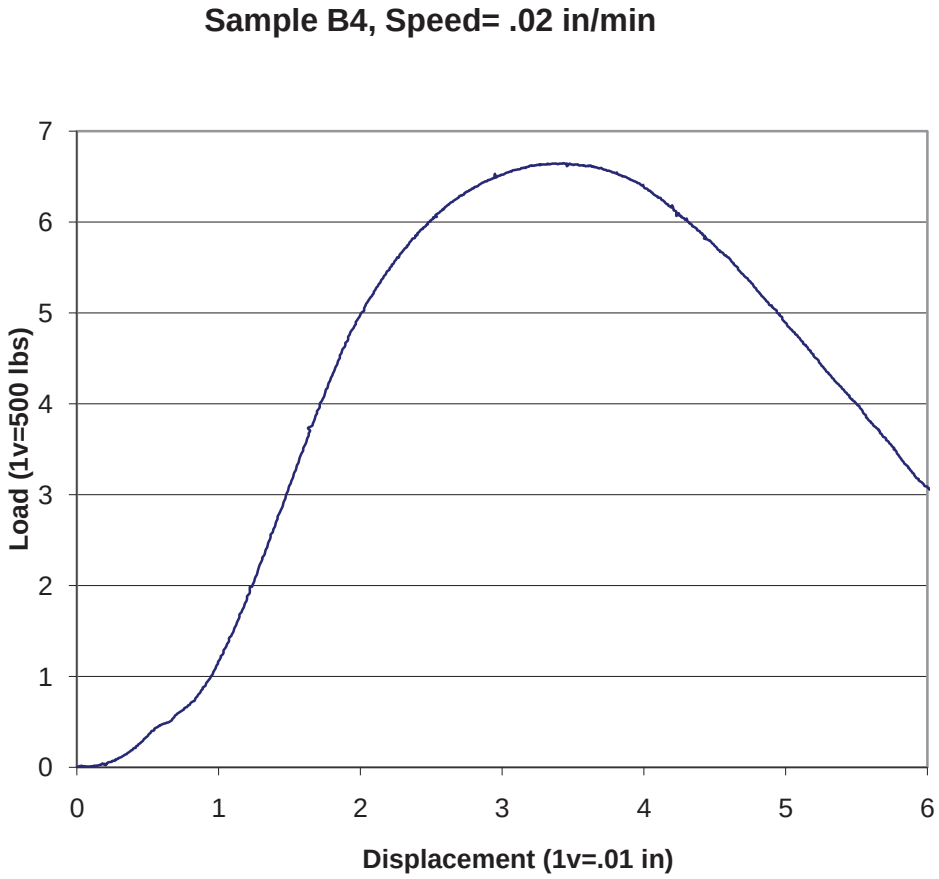
APPENDIX I: COMPRESSIVE STRENGTH DATA



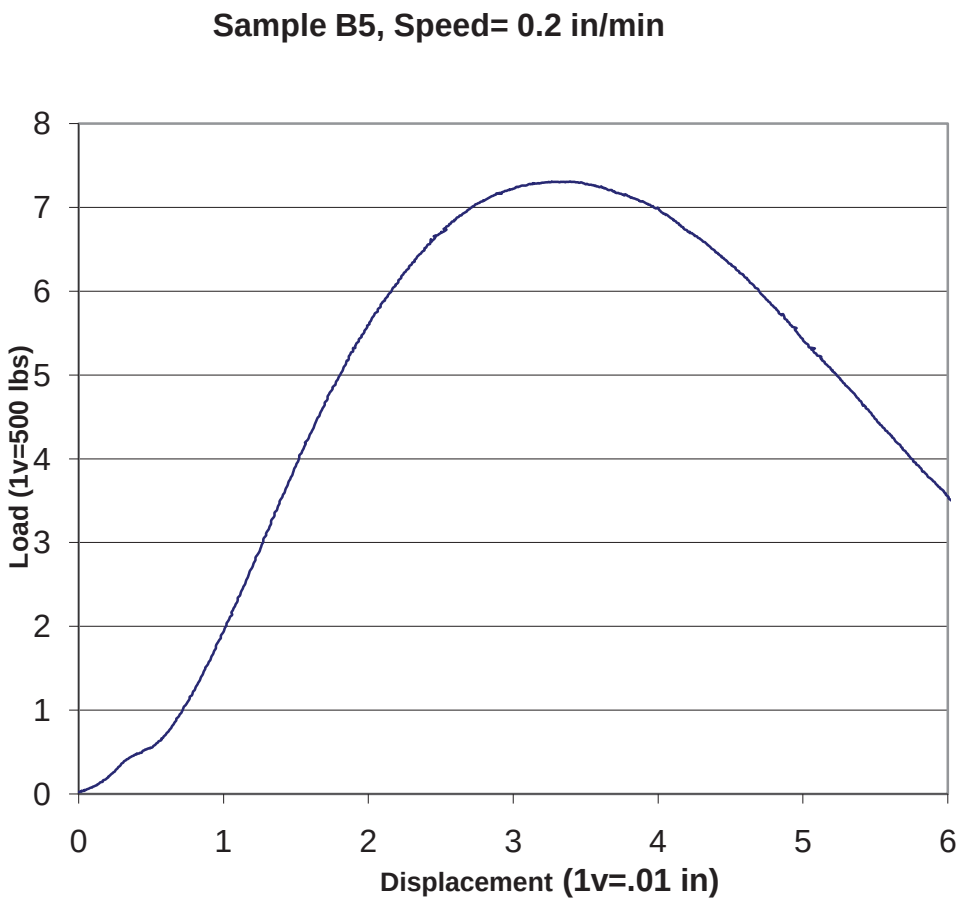
APPENDIX I: COMPRESSIVE STRENGTH DATA



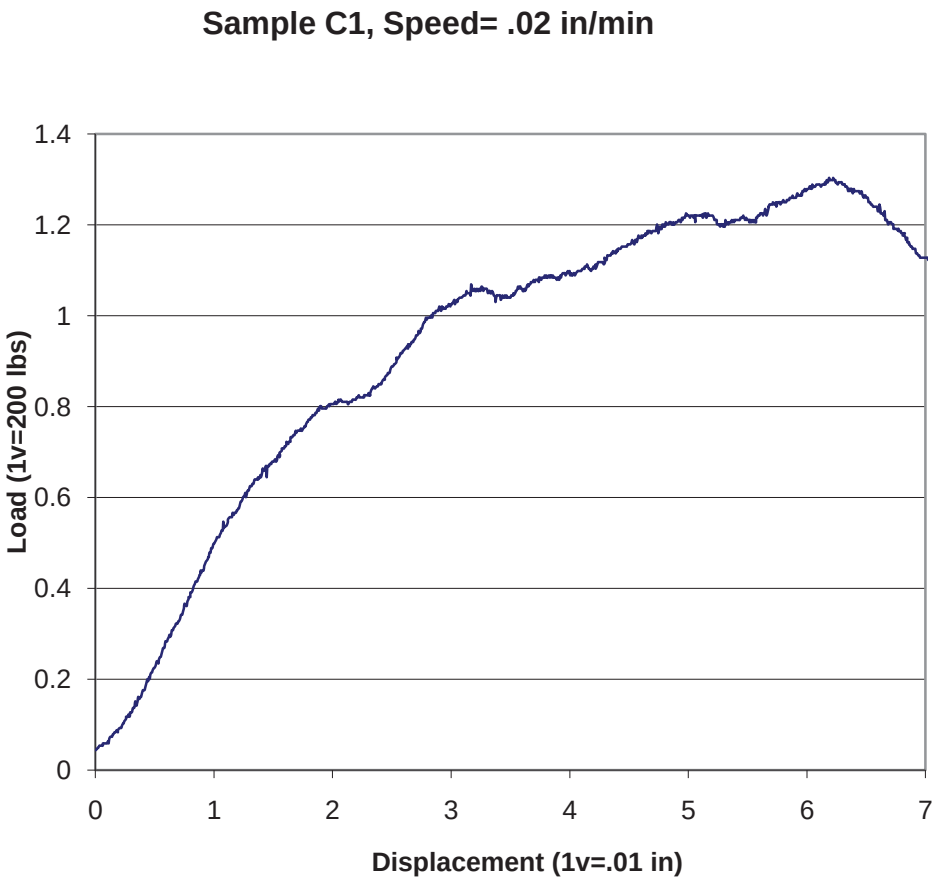
APPENDIX I: COMPRESSIVE STRENGTH DATA



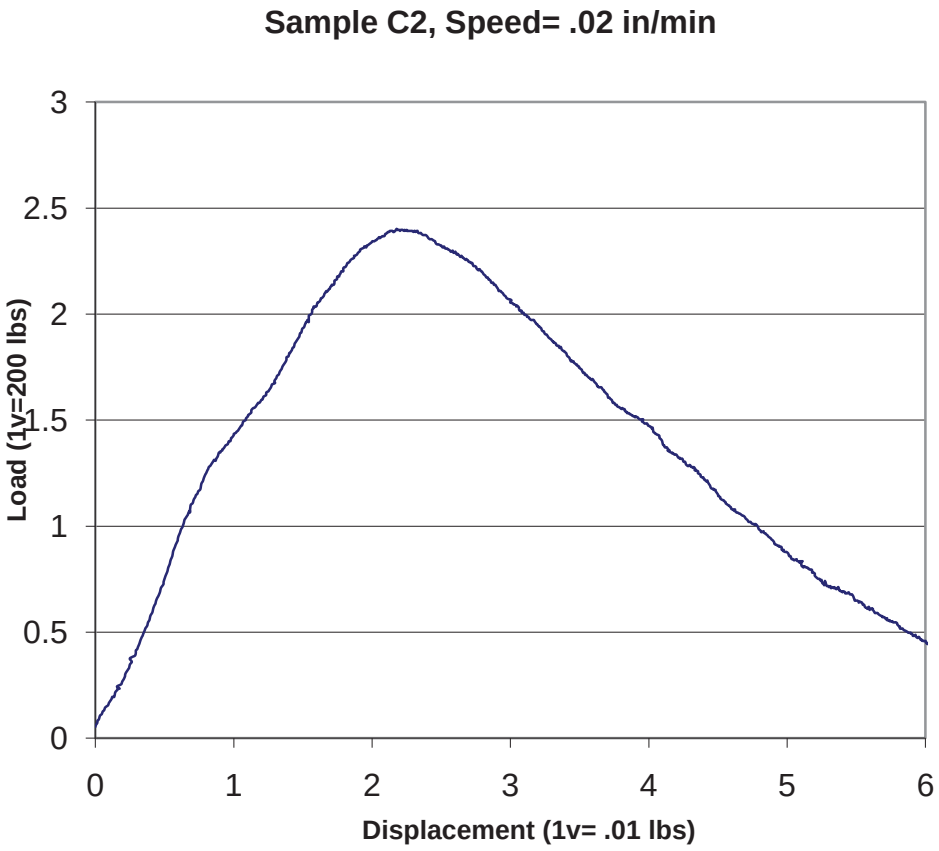
APPENDIX I: COMPRESSIVE STRENGTH DATA



APPENDIX I: COMPRESSIVE STRENGTH DATA



APPENDIX I: COMPRESSIVE STRENGTH DATA



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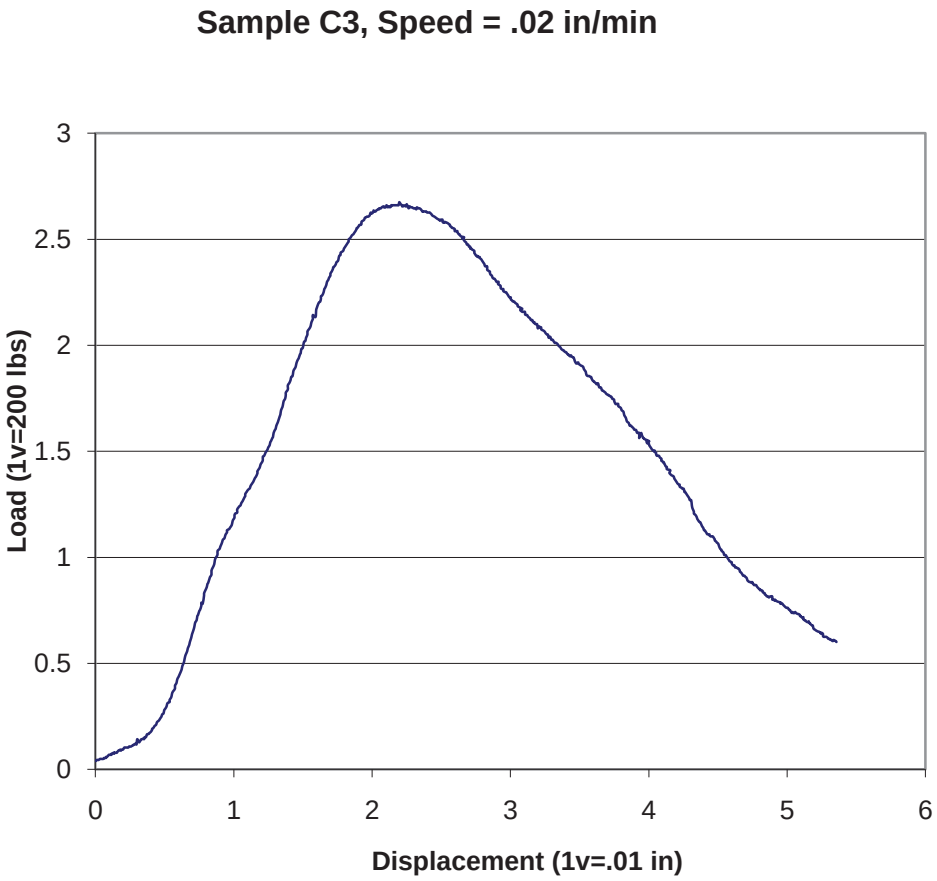

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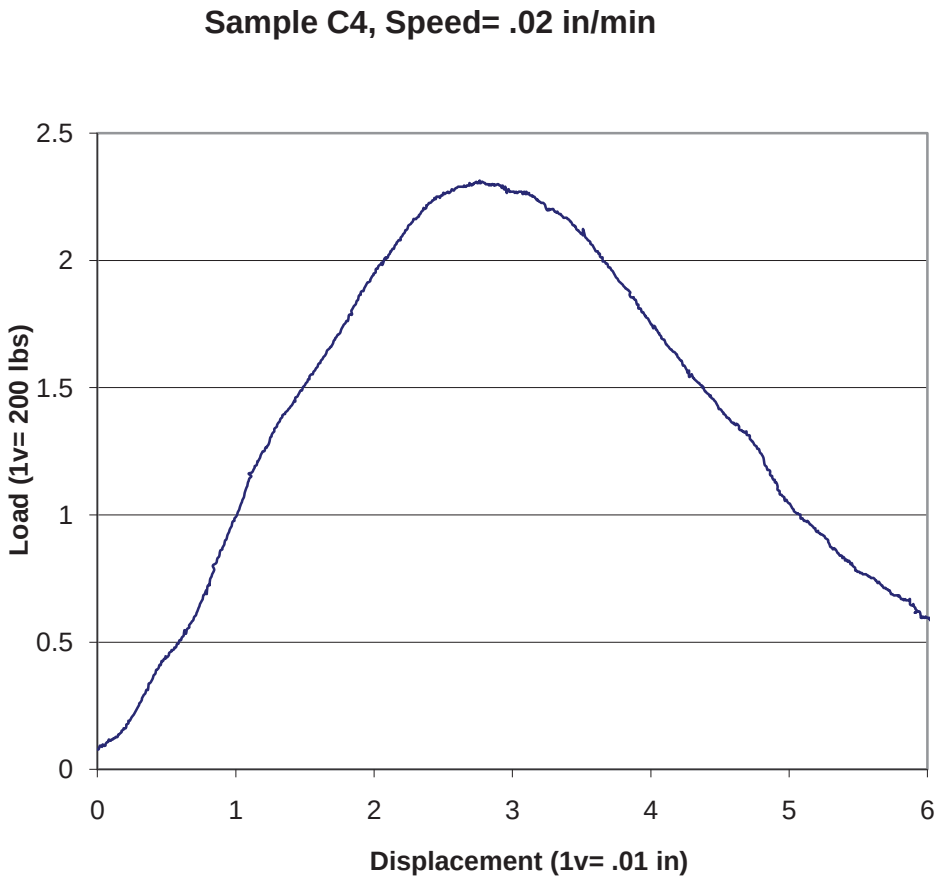

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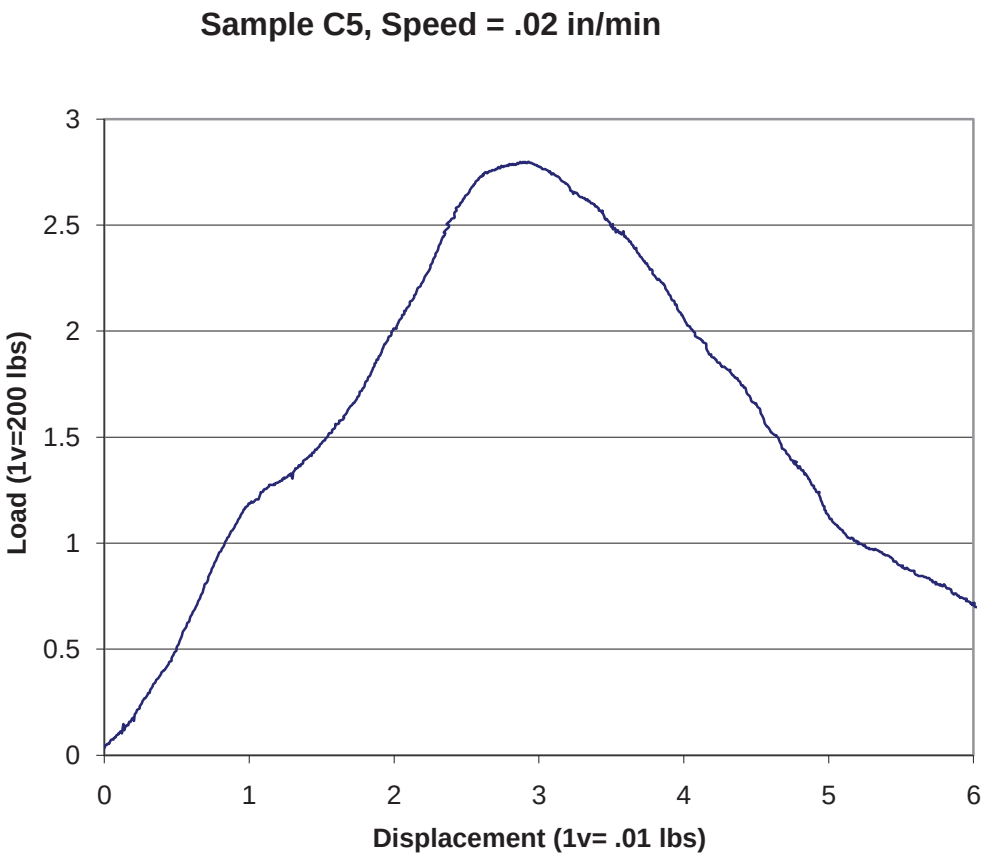
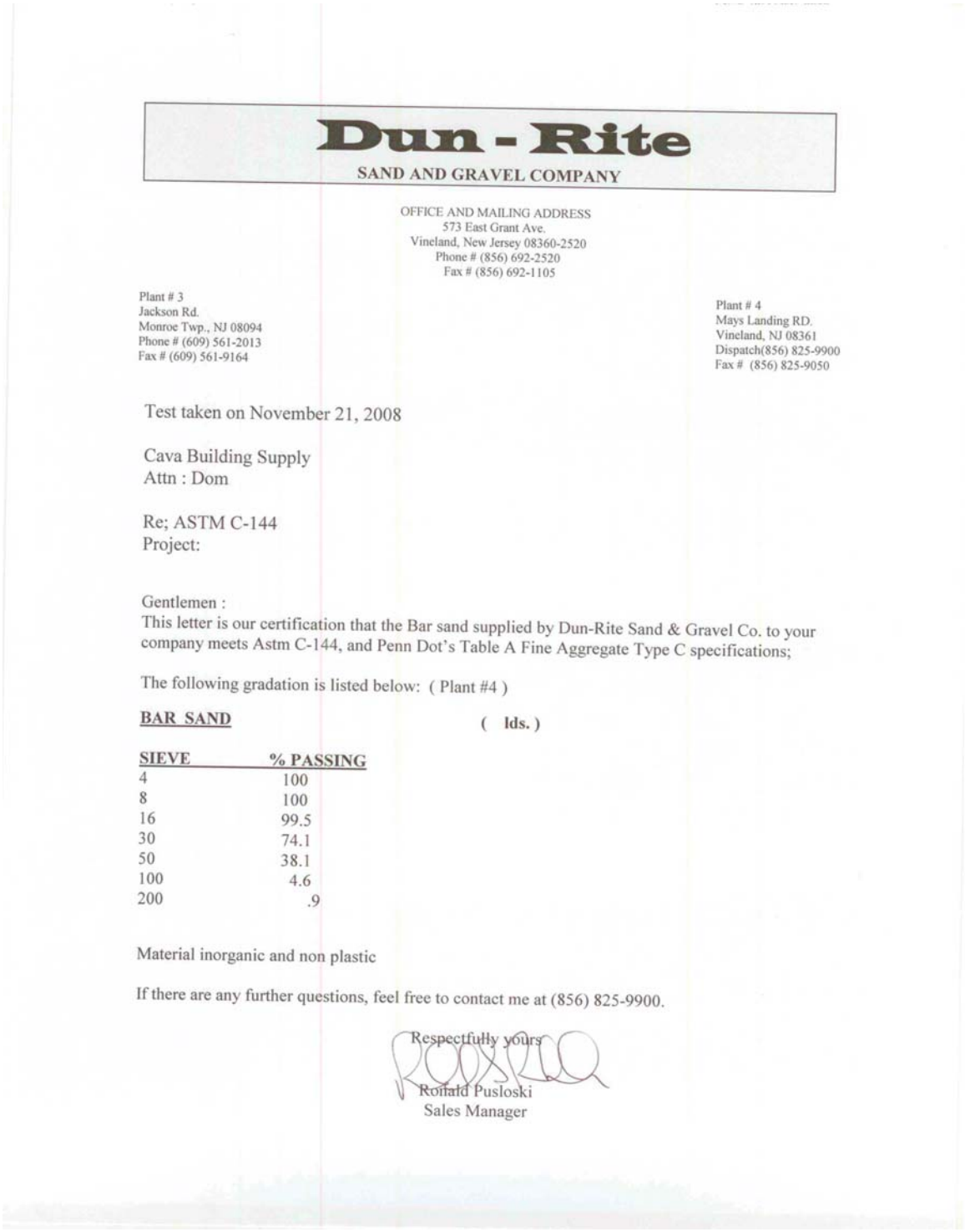


APPENDIX J: MATERIAL DATA SHEETS



Dun - Rite
SAND AND GRAVEL COMPANY

OFFICE AND MAILING ADDRESS
573 East Grant Ave.
Vineland, New Jersey 08360-2520
Phone # (856) 692-2520
Fax # (856) 692-1105

Plant # 3
Jackson Rd.
Monroe Twp., NJ 08094
Phone # (609) 561-2013
Fax # (609) 561-9164

Plant # 4
Mays Landing RD.
Vineland, NJ 08361
Dispatch(856) 825-9900
Fax # (856) 825-9050

Test taken on November 21, 2008

Cava Building Supply
Attn : Dom

Re; ASTM C-144
Project:

Gentlemen :
This letter is our certification that the Bar sand supplied by Dun-Rite Sand & Gravel Co. to your company meets Astm C-144, and Penn Dot's Table A Fine Aggregate Type C specifications;

The following gradation is listed below: (Plant #4)

BAR SAND **(lds.)**

SIEVE	% PASSING
4	100
8	100
16	99.5
30	74.1
50	38.1
100	4.6
200	.9

Material inorganic and non plastic

If there are any further questions, feel free to contact me at (856) 825-9900.

Respectfully yours

Ronald Pusloski
Sales Manager

APPENDIX J: MATERIAL DATA SHEETS

The National Brick Research Center
100 Clemson Research Blvd.
Anderson, SC 29625
(864)656-1094
www.brickandtile.org

2/8/2006

X-Ray Fluorescence Oxide Analysis

Submitter: Belden
Date: 08-Feb-06

Major Elements	Unit	MO3 Top	MO3 Middle	MO3 Bottom	MO6 Top	MO6 Middle	MO6 Bottom
Al2O3	%	31.11	25.06	24.77	32.65	27.03	24.93
SiO2	%	52.71	61.72	62.35	51.01	62.16	63.48
Na2O	%	0.33	<0.3	0.31	0.30	0.32	0.31
K2O	%	1.51	2.03	3.45	1.65	3.31	2.65
MgO	%	<0.10	<0.10	0.32	<0.10	0.36	<0.10
CaO	%	0.40	0.22	0.18	0.31	0.18	0.10
TiO2	%	2.15	1.51	1.38	2.08	1.35	1.38
MnO	%	0.00	0.00	0.00	0.00	0.01	0
Fe2O3	%	1.70	1.47	1.55	1.51	1.65	0.94
P2O5	%	0.09	0.07	0.08	0.08	0.09	0.07
S	%	0.03	0.03	0.05	0.10	0.03	0.03

Minor Elements	Unit	MO3 Top	MO3 Middle	MO3 Bottom	MO6 Top	MO6 Middle	MO6 Bottom
Cl	ppm	<50	<50	<50	<50	<50	<50
V	ppm	170	102	81	122	106	60
Cr	ppm	183	153	66	186	87	60
Ni	ppm	130	58	39	157	47	24
Cu	ppm	162	46	26	59	16	15
Zn	ppm	86	49	38	32	21	16
As	ppm	<4	<4	<4	<4	<4	<4
Rb	ppm	113	114	143	92	136	76
Sr	ppm	253	216	170	115	112	141
Y	ppm	170	163	153	175	141	116
Zr	ppm	347	250	322	361	342	390
Ba	ppm	801	545	786	456	740	464
Pb	ppm	<7	<7	<7	<7	<7	<7
LOI	%	9.73	7.42	5.36	10.12	3.34	5.97

Improved sulfur measurement is available by LECO SC-144 DR.
Analyses performed at The National Brick Research Center using a Noran QuanX EC Energy Dispersive Spectrometer provided by a gift from Ceric USA

Bedlen Brick Oxide Analysis

APPENDIX J: MATERIAL DATA SHEETS

58500 – 58580 Marble dust

58500	Marble dust, Italian, x-white, less than 32 µ
58520	Marble dust, extra fine grind, less than 32 µ
58540	Marble dust, medium grind, less than 90 µ
58560	Marble dust, coarse grind, less than 200 µ
58580	Marble dust, very coarse grind, 150 - 300 µ

Mineral Analysis

	58500	**58520**	**58540**	**58560**	**58580**	**58585**
$CaCO_3$ [%]	95.5	99.2	99.2	99.2	99.2	99.2
$MgCO_3$ [%]	3.0	0.4	0.4	0.4	0.4	0.4
FeO_3 [%]	0.08	0.035	0.035	0.035	0.035	0.035
Al_2O_3 [%]	-	0.1	0.1	0.1	0.1	0.1
SiO_2 (Silicates) [%]	-	0.25	0.25	0.25	0.25	0.25
Volatile content at 105°C DIN EN ISO 787-2 [%]	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Ignition loss DIN EN 459-2 [%]	43.6	43.8	43.8	43.8	43.8	43.8
HCl-unsoluble DIN 55 918 [%]	1.4	0.3	0.3	0.3	0.3	0.3

Physical Data

	58500	**58520**	**58540**	**58560**	**58580**	**58585**
Bulk density [g/cm^3]	0.76	0.75	0.73	1.0	1.2	1.35
Ramming density [g/cm^3] (DIN EN ISO 787-11)	1.4	1.4	1.35	1.7	1.6	1.65
Oil absorption [g/100g] (DIN EN ISO 787-5)	15	16	15	12	< 10	< 5
DOP-Value [g/100g] (nach DIN ISO 787-5)	25	28	27	17	12	10
Electr. conductivity (10%) [µS/cm] (DIN ISO 787-14)	62	43	40	43	46	38
pH-Value (DIN EN ISO 787-9)	9.4	9.6	9.6	9.6	9.6	9.6
Density [g/cm^3] (DIN EN ISO 787-10)	2.7	2.7	2.7	2.7	2.7	2.7
Hardness accor. To Mohs	3	3	3	3	3	3
Refraction index	1.59	1.59	1.59	1.59	1.59	1.59

Dr. Georg Kremer, Dipl.-Chemiker, Farbmühle, D-88317 Aichstetten/Allgäu, Telefon +49-7565-91120, Telefax +49-7565-1606
kremer-pigmente@t-online.de, www.kremer-pigmente.com

Marble Dust Data (Italian, x-white)

APPENDIX J: MATERIAL DATA SHEETS

Optical Properties

		58500	58520	58540	58560
uminosity (C/2°. DIN 53 163)		93.5	90.5	88	83.5
ellow value (DIN 6167)		3.5	7.6	10	13
Color index CIE AB (DIN 6174)		97.4	96.2	95.2	93.3
	a	0.1	0.6	0.4	-0.3
	b	1.8	3.7	5.2	7.2

Screen Analysis (DIN 53 734)

	58500	58520	58540	58560	58580	58585
Content of particles less than 630 µm						99.5 %
500 µm						94 %
315 µm					99.5 %	69 %
180 µm				99.6 %	88 %	30 %
90 µm			99.7 %	88 %	21 %	8 %
40 µm	99.9 %	99.8 %	97 %	59 %	9 %	
32 µm	99.6 %	99.6 %				

Particle Size Distribution (Laser-Granulometer)

	58500	58520	58540	58560	58580	58585
Content of particles less than 24 µm	97 %	98 %	91 %	47 %		
16 µm	88 %	91 %	83 %	43 %		
8 µm	66 %	70 %	64 %	34 %		
4 µm	45 %	44 %	42 %	22 %		
2 µm	26 %	25 %	21 %	13 %		

Mean particle diameter	4.5 µm	4.6 µm	5.1 µm	31 µ	130 µm	260 µm

Dr. Georg Kremer, Dipl.-Chemiker, Farbmühle, D-88317 Aichstetten/Allgäu, Telefon +49-7565-91120, Telefax +49-7565-1606
kremer-pigmente@t-online.de, www.kremer-pigmente.com

Marble Dust Data (Italian, x-white)

APPENDIX J: MATERIAL DATA SHEETS

CARMEUSE LIME, INC.

Annville Facilities

CARMEUSE
CHIMIE MINERALE

Typical Analysis of High Calcium Chemical Hydrate--Annville

Rev. #: 0	**Prepared By: Jeff Morrow**
Rev. Date: 10/31/02	**Approved By: Ed Hoffman**

Typical Chemical Analysis:

Based on 18 months of data from March 1, 2001 to August 31, 2002.
Data was obtained from hydrate production samples
Nominal sizing data is a best estimate and not supported by data. Sizing data is being developed to establish nominal sizing.
2σ control range for production.

CHEMICAL	AVERAGE (%)	MAXIMUM (%)	MINIMUM (%)	2σ
CaO	76.2	79.2	71.5	2.09
MgO	1.7	3.8	1.0	0.76
SiO2	1.1	1.5	0.6	0.26
S	0.03	0.07	0.01	0.018
Al2O3	0.4	1.0	0.2	0.14
Fe2O3	0.2	1.0	0.1	0.07
CO2	0.83	4.94	0.01	1.318
Available CaO	70.2	75.0	62.2	3.246
L.O.I.	24.1	26.3	21.3	1.19
H_2O	1.1	3.52	0.23	0.71
$CaCO_3$	1.81	7.19	0.02	2.564
$Ca(OH)_2$	92.7	99.00	82.10	4.298

Nominal Sizes Available:

Sieve Size	% passing
20 mesh	100.0
60 mesh	99.7
100 mesh	99.0
200 mesh	98.0
325 mesh	97.0

The information contained in this data sheet is, best of our knowledge, true and
---------- ›nal variations as the raw

COYNE CHEMICAL BL#: 895873 DATE: 02/16/2011
CUSTOMER: UNIVERSITY OF PENNSYLVANIA
PRODUCT CODE: QTY SHIPPED: 50
PO/RELEASE #: VERBAL-SARAH ROGERS
DELIVERY SITE/BLDG#: CUSTOMER PICK-UP
ORDERED BY: SARAH803-767-9460

›M on 1/3/03
.e--Annville

APPENDIX J: MATERIAL DATA SHEETS

CARMEUSE
NATURAL CHEMICALS

Carmeuse North America
11 Stanwix Street, 11th Floor
Pittsburgh, PA 15222
Phone: 412-995-5500
Fax: 412-995-5594

Date of Origin:	06/05/ 02
Date of Revision:	00 / 00 / 00
Revision No.	00

Material Safety Data Sheet

Product Name:	HYDRATED LIME

INFOTRAC: 800-535-5053 [In case of an emergency call this number 24 HOURS a day 7 DAYS a week.]

1. IDENTIFICATION OF THE SUBSTANCE AND COMPANY

1.1. Identification of the substance:

- Chemical name: Calcium hydroxide
- Product name: Hydrated Lime, High Calcium Hydrated Lime, Slaked Lime
- Formula: $Ca(OH)_2$
- C.A.S N°: 1305-62-0
- Molecular Weight: 74.08

1.2. Company:

Carmeuse North America
11 Stanwix Street, 11th Floor
Pittsburgh, PA 15222

Telephone: 412-995-5500
Fax: 412-995-5594

2. COMPOSITION / INFORMATION ON INGREDIENTS

Ingredient	% by Weight	C.A.S N°:	Exposure Limits
Calcium oxide	>68	1305-78-8	2 mg/m^3
Magnesium oxide	<4.5	1309-48-4	10 mg/m^3
Silica (Total)	<2.0	7631-86-9	0.1 mg/m^3 (respirable)

(This product may contain above >0.1% respirable silica dust)

3. HAZARD CLASSIFICATIONS

Irritant: To the eyes, mucous membranes and to the skin (in case of sweating).

Flammability: The product is not flammable or combustible.

Explosive: None

Reactivity: Reacts with strong acids produce heat and possible explosion in confined spaces.

Symbols: WHMIS Symbol: "E" Corrosive Material; "D2A" Materials causing other toxic effects.

4. FIRST AID SYMPTOMS AND TREATMENTS (Continued on page 2)

Effects:

- Inhalation: Irritations, sore throat, cough, sneezing.
- Eyes: Severe irritation, intense tearing, burns and possible blindness when exposure is prolonged.
- Skin: Removes natural skin oils, blotches, itching and superficial burns in case of sweating,
- Ingestion: Sore throat, stomach aches, cramps, diarrhea, vomiting.

APPENDIX J: MATERIAL DATA SHEETS

Product Name:	HYDRATED LIME (continued)

4. FIRST AID SYMPTOMS AND TREATMENTS . . . (Continued from page 1)

Treatments:

- Inhalation: Breath fresh air deeply. If necessary, give artificial respiration and seek medical attention.
- Eyes: Turn up eyelid and wash immediately and abundantly with lukewarm running water for at least 15 minutes. Do not try to neutralize. Obtain medical attention immediately.
- Skin: Remove contaminated clothes and rinse area with plenty of water.
- Ingestion: Drink water followed by diluted vinegar (1 part vinegar in 2 parts water) or fruit juice to neutralize. Do not induce vomiting. Obtain medical attention immediately.

5. FIRE FIGHTING MEASURES

- Classified as a non-flammable product.
- Decomposes itself into Calcium oxide at 580 °C. Calcium oxide is non-flammable, but generates heat in presence of water and acids.
- Safety measures in case of fire: avoid wetting lime, use appropriate extinguishing media to put out a surrounding fire.

6. ACCIDENT PREVENTIVE MEASURES

Individual and collective precautions:	Avoid contact with skin and eyes; wear waterproof shoes, gloves and safety goggles. Cover body with long sleeved shirt & long pants.
Avoid inhalation of dust:	Wear anti-dust mask.
Cleaning methods (Leaks & Spills):	Mechanical dry vacuuming; for small quantities, wash abundantly with water.
Precautions for the protection of the environment:	The substance may not be spilled without control into surface waters (increases pH).
Waste Disposal:	Dispose in secure landfill or according to federal, provincial/state and local environmental regulations.

7. HANDLING AND STORAGE

Handling:	In open air or in ventilated places. Avoid skin and eye contact. Avoid creating airborne dust. Store in dry places, sheltered from humidity.
Storage:	In waterproof bags or tanks. Keep away from acids and incompatible substances. Keep out of reach of children.

8. EXPOSURE CONTROL / PERSONAL PROTECTION

Technical Values:	Occupational exposure standard – 5 mg/m^3
Respiratory Protection:	Use a mask in unventilated dusty places.
Eye Protection:	Use safety goggles.
Hand Protection:	Use clean dry gloves.
Skin Protection:	Cover body with suitable clothes (long sleeves shirts and trousers covering the waterproof caustic resistant shoes).

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APPENDIX J: MATERIAL DATA SHEETS

Product Name:	HYDRATED LIME (continued)

9. PHYSICAL AND CHEMICAL PROPERTIES

• Physical State:	Solid
• Odor & Appearance:	Odorless, White powder
• pH:	12.5-12.8 at a concentration of 1.600 mg/l (saturated solution)
• Melting point:	Decomposes at 580° C to form calcium oxide
• Boiling point:	Decomposes at 580° C to form calcium oxide
• Flash point:	Non flammable
• Autoignition t°:	Non flammable
• Inflammability limits:	None
• Explosion risk	None
• Vapor pressure (+t°)	Non volatile
• Vapor density (air=ml):	Non volatile
• Relative density:	2.24 g/cm3
• Solubility:	Slightly soluble in water at 20°C: 1.65 g/l At 100°C: 0.71 g/l Soluble in acids, glycerin and sugar solutions

10. STABILITY AND REACTIVITY

• Stability:	Stable products, not very soluble.
• Decomposition temperature°:	580
• Reactivity:	Reacts with acids to form calcium salts while generating heat
• Conditions to avoid:	Wet places, vicinity to incompatible materials
• Incompatible materials:	Water; acids; reactive fluoridated, brominated or phosphorous compounds; reactive powdered metals; organic acid anydrides; nitro-organic compounds; interhalogenated compounds
• Hazardous decomposition products:	None

11. TOXILOGICAL INFORMATION

Toxicity:	Non-toxic substance.
Exposure Limits:	TWAEV – 5 mg/cu.m
Other:	Respirable SiO_2, may be a component of this product and is classified by IARC as a group 1 human carcinogen. No reported Carcinogenicity, Reproductive Effects, Teratogenicity or Mutagenicity.

12. ECOLOGICAL INFORMATION

- Alkaline substance that increases pH to 12.5 at a concentration of 1.60 mg/l.
- Calcium hydroxide shall progressively be rendered soluble and shall recarbonate to form calcium carbonate (CaC03).
- Calcium carbonate is ecologically neutral.
- Uncontrolled spillage in surface waters should be avoided since the increase pH could be detrimental to fish.

- 2 -

Product Name:	HYDRATED LIME (continued)

13. DISPOSAL CONSIDERATIONS

- Dispose in secure landfill or according to federal, provincial/state and local environmental regulations.

14. TRANSPORTATION INFORMATION

Classification:	TDG	Not listed for ground transportation
	HMR	Not listed for ground transportation

TDG: Transportation of Dangerous Goods Regulation (CAN)
HMR: Hazardous Materials Regulation (USA)

15. REGULATORY INFORMATION

Symbol:	E	Corrosive Material
	D2A	Materials causing other toxic effects
Risk Phases:		Risk of serious damage to the eyes.
Safety Phases:		Keep out of reach of children. Keep storage container away from humidity. Avoid contact with skin. Avoid contact with eyes. In case of contact with eyes, rinse immediately with water for at least 15 minutes.

16. MISCELLANEOUS OTHER INFORMATION

GLOSSARY OF TERMS

Calcination: the firing of materials at high temperatures (Torraca, 2009, p. 50)

Carbonation: process by which lime mortar reabsorbs carbon dioxide and reverts to calcium carbonate, leaving mortar harder, more stable, and less soluble (Gibbons, 2003, p.61)

Cementitious: property of a material that sets and develops strength through a chemical reaction with water in which hydrates are formed in a reaction that is capable of occurring under water (ASTM C219, 2001, p.2)

Cocciopesto: Italian mortars and plasters made with hydrated lime and powdered brick (Bugini, 1993, p. 386)

Hydrated calcium aluminate: product of pozzolanic reaction resulting from reaction of amorphous alumina with lime that contributes to hydraulic set (Torraca, 2009, p. 55)

Hydrated calcium silicate: product of pozzolanic reaction resulting from reaction of amorphous silica with lime that contributes to hydraulic set (Torraca, 2009, p. 55)

Hydrated Lime: type of lime in which the quicklime has been slaked with just enough water to form calcium hydroxide in the form of dry powder (Gibbons, 2003, p. 62)

Hydraulic Lime: the hydrated dry cementitious product obtained by calcining a limestone containing silica and alumina, or a synthetic mixture of similar composition, to a temperature short of incipient fusion so as to form sufficient free lime (CaO) to permit hydration and at the same time leaving unhydrated calcium silicates to give the dry powder its hydraulic properties (ASTM C219, 2001, p. 2)

Lime putty: hydrated lime which has been slaked from quicklime using sufficient water to form a thick liquid and subsequently settled out to a putty during storage (Gibbons, 2003, p. 62)

Lime water: a saturated solution of calcium hydroxide in water left when lime putty settles out of slaked lime (Gibbons, 2003, p. 62)

Pozzolan: a siliceous or alumino-siliceous material that in itself possesses little or no cementitious value but that in finely divided form and in the presence of

moisture will chemically react with alkali and alkaline earth hydroxides at ordinary temperatures to form or assist in forming compounds possessing cementitious properties (ASTM C593, 2000, p. 1)

Pozzolana: a soil found near the Roman town of Pozzouli formed by the deposition of volcanic ash and containing silica and alumina that, upon rapid cooling, forms some crystalline silica-aluminates as well as amorphous glassy particles (Torraca, 2009, p.54)

Pure Lime (high calcium lime): Lime derived from limestone with less than 5% magnesium carbonate (Elert, 2002, p. 62)

Slaking: the controlled process of combining quicklime with water to form lime putty or hydrated lime (Gibbons, 2002, p. 63)

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