Contractor Report

Ancient Cementitious Materials

Waste Isolation Pilot Plant
Carlsbad, New Mexico

August 31, 2000

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1.0 Introduction

The U.S. Department of Energy (DOE) is initiating a program of passive institutional controls (PICs) for the Waste Isolation Pilot Plant (WIPP). This program is required by U. S. Environmental Protection Agency (EPA) regulations 40 CFR 191.14(c) (EPA, 1993) and 40 CFR 194.43 (EPA, 1996). The primary purpose of the PICs program is to indicate the location of the repository and its dangers, thus reducing the likelihood of inadvertent human intrusion into the repository.

A preliminary design for systems to mark the site has been developed. It includes five new components:

1. Large Surface Markers;
2. Small Subsurface Markers;
3. Berm;
4. Buried Storage Rooms;
5. Information Center.

The current design calls for granite to be used as the primary material of construction for the large surface markers, small surface markers, buried storage rooms, and information center (DOE, 1999a).

Although the reference design specifies granite, it has been suggested that concrete may be an acceptable alternative material of construction (Sandia National Laboratories, 1993; Mehta, 1999). Accordingly, a literature review has been performed to investigate instances in which man-made cementitious materials have survived for very long time periods. The intent of this effort is to determine and document, when possible, the attributes of cementitious materials that allow them to survive for long periods. It is also intended to illustrate that cementitious materials were used as effective building materials thousands of years ago and many of those materials have lasted throughout the centuries.

Section 2.0 provides a historical overview of the use of cementitious materials. Section 3.0 provides case studies specifically compiled for this report. Finally, Section 4.0 provides conclusions based upon the literature review information.

2.0 History of the Use of Cementitious Materials

A historical overview of the use of cementitious materials is provided in this section. The information is broken into three era-specific sections, based upon information found in the literature review. Greater detail on the specific examples of the use of cementitious materials is presented in Section 3.0.

2.1 Pre-Roman Cementitious Materials

Pre-Roman cementitious materials primarily consist of lime mortars between approximately 3,000 and 9,000 years old. Greek concretes are also classified as pre-Roman cementitious materials.

One of the earliest documented examples of the use of a cementitious material occurred nearly 9,000 years ago in the form of a floor slab. This floor slab was constructed using a large quantity of pyro-processed lime and resulted in a structure with a strength comparable to that obtained in modern structural concrete. Another example is the mortar used in construction of the Egyptian pyramids. This mortar was used to fill cracks and voids; it significantly enhances structural properties of the pyramids.

The Greeks appear to have been the actual discoverers and first users of pozzolanic concrete (i.e., concrete using volcanic ash as a constituent).[1] In one of the earliest examples of the use of concrete, the Greeks constructed a water tank that still stands today, after 3,000 years of weathering, and still maintains admirable physical properties.

2.2 Roman Cementitious Materials

The Romans used concrete extensively throughout their empire, particularly pozzolanic concretes, between approximately 1,500 and 2,000 years ago. Many substantial Roman structures made of concrete including roads, buildings, aqueducts and seaports have survived nearly 2,000 years to present times. Unfortunately, the concrete capabilities of the Romans appear to have ceased with their empire and were not resumed, to a large extent, for nearly 1,300

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[1] Pozzolan is an important concept in regard to high-performance (including high-durability) concretes. The term pozzolan is derived from the city Pozzuoli, near Mount Vesuvius, Italy, where one of the first pozzolans was discovered. It refers to a chemical reaction that allows silica combined with lime to form a solid cementing material. This chemical reaction is driven by the addition of the pozzolanic material. While the original pozzolanic material was volcanic ash, modern pozzolans include materials such as fly ash and silica fume, which are industrial byproducts.

years.

Steiger (1995a), provides a brief history of the development of concrete with particular attention paid to Roman concretes. In his discussion of Roman concrete, the author addresses the first examples of Roman concrete dating to 300 B.C. and the discovery of pozzolanic concrete:

> Roman concrete, although unreinforced, still stands today and was used on a large scale. Both the Colosseum (completed in A.D. 82) and the Pantheon (completed in A.D. 128 . . .) contain large amounts of concrete. The Basilica of Constantine and the foundations of the forum buildings also were constructed of concrete.
>
> In the Pantheon, aggregate in the concrete ranged from heavy basalt in the foundations and 20-foot-thick lower walls, through brick and tuff (a stone formed from volcanic dust) in the upper walls, to the lightest of pumice toward the top of the 142-foot diameter dome. The entire structure is in good condition and stands today in original form. It is an amazing feat considering that the builders did not understand the chemistry of hydraulic lime.
>
> Unfortunately, although the Romans used concrete in many structures built across their far-flung empire, it was impractical to transport volcanic ash from Italy. Therefore, the pozzolan technology used so successfully in Rome, did not survive the fall of the Roman Empire in A.D. 476. All concrete construction for the next 1,300 years used lime mortars and concretes.

2.3 Post-Roman Cementitious Materials

Post-Roman cementitious materials include both current concrete formulations and those up to approximately 900 years old. Although concretes of Mesoamerica ("[A] term for the culture area occupied by the native peoples of central and southern Mexico, Guatemala, Belize, and western Honduras and El Salvador" [Grolier, 1998]) are not as well documented as Roman concrete, there are fine examples of post-Roman pozzolanic concretes that appear to be as durable, if not more durable, than Roman pozzolanic concretes. For example, samples of Mayan concrete from approximately 900 years ago were tested and the results compared against the performance of Roman concretes. The data indicate that the Mayan concretes have substantially greater freeze/thaw durability than do the Roman concretes (Brown, 1987).

Also of interest is the recent construction of a temple in Hawaii. The construction specifications for this project required the use of a concrete designed to last a minimum of 1,000 years. The engineer responsible for specifying concrete for the project studied ancient concretes. He decided to formulate a modern pozzolanic concrete that does not utilize steel reinforcement. It appears that modern concretes have durability properties comparable to those of the ancient concretes.

3.0 Case Studies

The most pertinent case studies were selected during the literature review process and are presented in this section. To the extent appropriate, the descriptions follow the chronology established in Section 2.0.

3.1 Evolution of Cementitious Materials

Bonen, et al. (1994), provide an analysis of the earliest known uses of cementitious materials along with analyses of the use of cementitious materials through approximately 500 A.D. The authors attempt to determine if concrete durability is correlated with some physical property such as high strength, or alternatively, whether durability is a function of the thermodynamic stability of the emplaced cementitious material.[2] The following sections describe case studies performed by the authors and summarize their conclusions.

3.1.1 Lime Plastered Floor in Israel

One of the earliest cited uses of a cementitious material is a lime plastered floor found in the village of Yiftahel in Israel (Bonen, et al., 1994). Carbon-14 age dating of seeds found within the floor, covering an area of over 65 m^2 and weighing approximately 1.6 tons, indicated that its age is over 8850 years, plus or minus 50 years. The floor typically consists of two layers with a base layer approximately 45 mm thick and a 5mm finish layer. Through X-ray analysis it was determined that the primary constituents of both layers is calcite (formed by carbonation of lime) with minor amounts of quartz. The authors note that the upper finishing layer exhibits greater strength properties which is likely a result of some type of mechanical compaction.

Through an analysis of specimens taken from this and other lime plastered floors found throughout Israel, it was determined that the floors exhibit a compressive strength of 34 Mpa (millipascals) for the base layer and a compressive strength of 45 MPa for the finish layer. The authors note that while this floor does not contain aggregate like modern structural concrete, its compressive strength is comparable to modern structural concrete.

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[2]Thermodynamic stability refers to the movement (i.e., expansion and shrinkage) of concrete that results from the initial heat generated through the chemical reaction that causes the materials to bind together and the subsequent loss of that heat until the product reaches ambient temperature. A product exhibiting a larger degree of thermodynamic stability would have a smaller temperature spike when the product was emplaced.

3.1.2 Pyramid Mortars

A review of ancient mortars was also performed by Bonen, et al. (1994). The authors discuss the use of a calcium sulfate-based mortar as a cementitious material and as a filler for cracks and open spaces in the Egyptian pyramids. The main constituents of this calcium sulfate-based material are gypsum, anhydrite, calcite, argillaceous limestone, and quartz sand. Based upon the authors review of previous investigations, it was determined that when the quarried gypsum bed rock was heat treated, it possessed pozzolanic properties due to the breakdown of clay to amorphous aluminosilicates.

The authors also learned through their own literature review that it has been proposed that the mechanical integrity of the Pyramids is directly related to the performance of the mortars. They found that the collapse of the Meidum pyramid built in 2600 B.C. was due to the poor quality of the binding material which was composed of argillaceous materials and large gypsum grains. The pyramids that were built in 2500 B.C. and later, however, have stood intact as a result of the superior binder with finer anhydrite grains. The cementitious materials in these stronger pyramids have lasted approximately 4500 years to date.

3.1.3 Roman Cementitious Materials

Bonen et al. (1994) also discuss the concrete era tracing the use of concrete as far back as 2300 years ago. In particular the Roman's development and use of lime-pozzolanic concrete marks what is referred to as the concrete era[3]. The authors expressed a specific interest in the performance of Roman concrete structures that were, and continue to be, continuously subjected to salt attack. They cite the City of Caesaria in Israel on the shores of the Mediterranean which was built around 10 B.C. In describing the manner in which concrete was used in the construction of the city, Bonen et al. (1994) state:

> Among the architectural innovations was the building of a port enclosing over [more] than 200,000 m^3 of seawater. It was developed by building a breakwater about 600 m long and 50 m wide made of two external walls and a concrete filling. The outside wall facing the sea waves was made of large stone up to 15 x 3 x 3 m in size that were attached to each other by lead clamps. Following the construction of the walls, the space between them was filled with concrete made of fieldstone, rubble, red clay, and lime.

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[3]For purposes of this literature review "concrete era" refers to the time period beginning with the Roman's first use of concrete, approximately 2300 years ago, to present times. This is not to say there are no fine examples of concrete existing before this era, but rather that the literature tends to give the Romans credit for first implementing the wide-spread use of concrete.

The famous Roman aqueducts are also cited as exhibiting excellent physical properties in light of the fact that they were built nearly 1800 years ago and some of them are located as little as 150 meters from the sea and are continuously sprayed with sea salts and submitted to cycles of wetting and drying.

3.1.4 Basis for Physical Properties

Finally, the case study authors cite analyses and draw conclusions regarding the potential reasoning for some of the ancient mortars and concretes ability to survive over such great expanses of time. As Bonen et al. (1994) recognize ". . . it is a well accepted that permeability is the single most important factor affecting the durability of portland cement concrete . . ." As a result, they conducted permeability tests on samples of ancient concretes. The test results on a sample of approximately 1400-year-old khorasan mortar from Instanbul, Turkey, indicate that the sample was relatively permeable for concrete (K-10^{-6} cm/sec; K being a permeability factor) and had a relatively high porosity (water absorbency of 30 to 45 percent). The authors also note that even though the ancient concrete exhibits permeable characteristics, it has fared extremely well as compared to modern concretes. The authors state that ". . . in contrast to portland cement concrete that has been a few years in service, only a few microcracks were observed in these khorasan mortars."

Given these observations and other chemical analysis, the authors conclude with the following remarks:

> Portland cement concrete is distinguishable by its high rate of strength development and superb mechanical properties as compared to lime concrete. However, it appears that the Achilles' heel of portland cement concrete is it[s] susceptibility to environmental attack. In turn, the outstanding performance of the lime concrete is neither attributed to its compressive strength nor to its impermeable nature. Rather the durability of ancient concrete is related to the higher thermodynamic stability of its alteration products.

3.2 The Ancient Kamirian Water Storage Tank: A Proof of Concrete Technology and Durability for Three Millenniums

Probably the best example of pre-Roman concrete exists in the Greeks use of concrete. Koui (1998), provides an analysis of a concrete water tank found in the ancient city of Kamiros on the Greek island of Rhodes. The water tank was constructed approximately 3000 years ago and remains intact today. The research focused on the physical, chemical, and mechanical tests carried out on a concrete sample taken from the water tank. The test results illustrate the concrete's significant durability. They also support a conclusion that the ancient Greeks had an excellent knowledge of concrete technology three millenniums ago.

An analysis of the water tank sample provided researchers with much information. The researchers were able to determine the current compressive strength of the specimens and accounting for the fact ". . . that the compressive strength curve development has passed through its maximum value centuries ago . . " they concluded that the ". . . compressive strength continues to be at amazingly high levels." In addition, they were able to determine through compression tests ". . . that the mass of this concrete exhibits good elastic properties." The researchers also performed water permeability tests and porosity measurements, which revealed ". . . that the porosity of the ancient concrete is amazingly low, compared to that of a similar contemporary concrete . . . while its water-tightness is quite satisfactory despite the dissolving effect of the rainwater and of the activity of humic acids over about 3 millenniums." The researchers attribute the favorable results as follows:

> The low porosity and the good water tightness of the concrete, of course, are attributed to the excellent choice of the aggregate grading and the binding material, which in turn resulted in excellent durability behavior of the surviving concrete of ancient Kamiros.

After investigating the types and amounts of aggregates and the binder used, the researchers offer the following conclusions regarding the water storage tank of Kamiros:

> . . . it is concluded that a mixture of aggregates consisting of siliceous gravel, granular intermediate calcareous aggregates and fine-grained calcareous aggregates had been used, in such proportions as to produce an ideal total gradation of the blend and volcanic earth, with lime as a binder.
>
> The technology used for the concrete construction and the resulting excellent physical and mechanical properties of the concrete found after three millenniums of weathering reveal the excellent "know how" the ancient Greeks had in this field.

3.3 Ancient Structural Concrete in Mesoamerica

While it would appear from a number of the available sources that ancient structural concrete was used primarily in Europe and Asia, there are citations referencing the use of structural concrete in Mesoamerica between 900 and 1150 years ago. Rivera-Villarreal (1996), investigated the use of structural concrete in Mesoamerica and how that concrete has survived over the centuries.

Rivera-Villarreal performed an analysis upon a core sample of a concrete roof that was found in 1991 during the most recent restoration of the ancient city of El Tajin in Mexico. Although the roof was cracked, which was determined after the removal of trees; vegetation; and soil; it was complete. A series of tests were then performed on a sample of the uncovered concrete to determine the physical properties of the roof slab.

Chemical analysis of the roof slab showed that the main components were calcite and quartz; also found were diopside and nepheline composites, which result from pozzolanic reactions. The author determined that ". . . the cementitious composition included: 15 percent soluble silica, 15 percent aluminum, 56 percent lime (the major component) and 20 percent anhydrous cementitious material." He notes that this composition ". . . is similar to natural cement, with a pozzolanic index of 0.42, indicating that silica and alumina mobilization in the cementitious material is quite significant." Thus, the concrete composing the roof slab is what is commonly referred to as a pozzolanic concrete.

Unfortunately, it was impossible for the author to determine the age of the roof at the time of its collapse. The author does speculate, however, that the roof had remained functional for many years and probably collapsed as a result of seismic action. Nevertheless, the concrete roof slab pieces remained intact after the collapse.

3.4 A Comparison of Ancient Roman and Mayan Concretes to Modern Concretes

To address the question of freeze-thaw durability of ancient Roman concrete, Brown (1987) tested two samples of Roman concrete approximately 1900 years old, two samples of Mayan concrete from Mexico approximately 600 years old, and a reference sample of modern-day concrete. The samples were subjected to freezing in air and thawing in water and the weight losses were recorded.

The author found that the Roman concretes virtually disintegrated after 18 and 29 cycles respectively of freeze-thaw conditions. Interestingly, however, one of the Mayan samples lasted for 75 cycles while the other Mayan sample survived the entire test of 150 cycles of freeze-thaw conditions with 28 percent loss. The author speculates that the 1300-year age difference had no ". . . appreciable effect on the reduced durability found in the Roman concretes." The reference modern-day sample survived the entire 150 cycles with only six percent loss. From these tests the author surmises:

> Although the bridges and parking structures of today do have their shortcomings, modern concrete is vindicated somewhat by these tests. Although the Colosseum has withstood the test of 2000 years in the benign climate of Italy, it would likely have survived only a few years under the severe weather conditions of Canada.

The author states clearly, however, that he was not concluding that Roman concrete was an inferior product, rather, he states:

> By using pumice, a natural lightweight aggregate, the Romans were the first to create lightweight concrete. The arches in the Colosseum contain lightweight concrete as does the 142-foot roof span of the Pantheon in Rome. The Pantheon

> roof was formed and poured, similar to a modern day concreting operation. And after 2000 years it is still open for inspection!
>
> Mile-long arched aqueducts were built without expansion joints. Although exposure to wet and dry seasons and temperatures between 32 degrees F and 95 degrees F do create considerable deformations in many examples of concrete, the aqueducts performed without cracking. Even the linings are free of shrinkage cracks. The use of oil admixtures and particular attention to the troweling of those linings, layer after layer, took a great deal of skill. Many of the aqueducts are still in use today.

Brown concludes that while the Roman concrete could not withstand a rigorous freeze-thaw test, it was more than adequate for the climate in which it was developed.

3.5 Roads of the Roman Empire

The roads of the Roman empire consisted of ". . . nearly 53,000 miles of roads linking the capital to their far-flung empire." By comparison, in 1995 there existed 42,000 miles of interstate highways in the United States. Steiger (1995), provides an analysis of the Roman's use of concrete as a building material in their road system which was constructed over 1500 years ago and survives, in part, today.

In short, Roman roads typically consisted of four courses presented in ascending order as follows:

1. A statumen layer of large, flat stones 10 to 24 inches thick;
2. A radius course, about 9 inches thick, consisting of smaller stones mixed with lime;
3. A nucleus layer, about 1 foot thick, consisting of Roman concrete made with small gravel and coarse sand mixed with hot lime and water;
4. A summa crusta, or wearing surface of flintlike lava stones, fitted tightly, about 6 inches thick.

The third layer, the nucleus layer, is the layer of interest in this report. Steiger notes that the concrete mix, whenever possible, consisted of lime mixed with volcanic rock or sand. In describing the pozzolanic reaction Steiger states:

> The pozzolana contained an aluminum silicate from which silica was readily liberated by caustic alkalies, such as calcium hydroxide. Silica combined with the lime to form a solid cementing material that would harden in water.

In agreement with the case study by Brown in the previous section, Steiger concludes that lime-silica mortar in conjunction with favorable climatic conditions provides the basis for the amazing endurance of many of the ancient Roman roads.

3.6 The Thousand Year Temple in Hawaii

In a recent application of ancient concrete technology, a massive unreinforced concrete slab was poured in Hawaii. This recent application of ancient concrete technology is important to an analysis of ancient concrete's ability to survive over such long periods. This case study can be best summarized by Dr. P.K. Mehta's quote captured in a press release relating to the concrete's application:

> "This hasn't happened for 2,000 years!" chortled Dr. P.K. Mehta. "Its historic. Not since the Greeks and Romans has such a massive placement of concrete been completed without a single crack. Not even a hairline fissure." (Press Release, 1999)

The structure referred to by Dr. Mehta is a 117 foot 6 inch long by 56 foot wide by 4 foot thick temple foundation that weighs over 4 million pounds taking 108 cement trucks to place and has no reinforcement. The foundation was specifically designed to last for a minimum of 1,000 years. It will be required to carry a load of ". . . four million pounds of carved stone without cracking, nor sink more than 1/8th of an inch over a twelve foot span--ever--or else the temple's long granite roof beams would break and fall" (Mehta, 1999).

Dr. Mehta specifically ruled out the use of reinforcing steel as experience indicates that while such steel reinforcement allows for stronger concretes over a shorter period of time it also promotes deterioration of that same concrete within 20 to 40 years. That is, while steel reinforcement tends to prevent large cracks, small cracks develop, which, in turn create environmental pathways that corrode the steel and destroy the concrete (Mehta, 1997). In order to address these concerns it was necessary to develop a concrete that requires no reinforcement.

High volume fly ash concrete was offered as a solution by Dr. Mehta. This is a type of concrete in which half or more of the cement in the mix is replaced with fly ash, a waste byproduct of coal-burning electrical plants. Dr. Mehta stated:

> The ancient Romans and Greeks knew how to make something very similar to modern concrete using volcanic ash form the Italian town of Pozzuoli. They built structures such as the Pantheon, whose 140-foot diameter concrete dome stands to this day (Mehta, 1999).

Dr. Mehta goes on to note that while volcanic ash is not used in modern concrete there would be some substantial benefits to doing so for the right applications. One of these benefits is that concrete using fly ash ". . . ultimately is stronger than normal concrete of equivalent design . . . ."

Fly ash concrete is also “. . . much more durable than normal concrete because it can almost totally eliminate temperature cracking due to its low heat of hydration and slow cure time (90 days).” Another substantial benefit is that this type of concrete does not require the steel reinforcement that aids in the deterioration of concrete. Also, the use of fly ash allows for the use of a waste product in place of a product (portland cement) that when produced causes large amounts of carbon dioxide.

Dr. Mehta explains that fly ash concrete has a longer curing time. The concrete slab reached 1,000 psi in three days and 3,000 psi in three weeks. Dr. Mehta also notes that the slab strength is expected to reach 6,000 psi in a few years and that fly ash concrete continues to gain strength “. . . longer than equivalent mixes using cement only.” And while it will not reach South Indian granite bedrock strength at 16,000 psi, Dr. Mehta stated that it was “. . . close enough . . .” (Press Release, 1999).

Dr. Mehta explains that it is important to develop a specific mix for the locality:

> Mehta first came to Honolulu to do dozens of trial mixes . . . . After the results of this were in, he refined the formula further with additional trial mixes. Each ingredient had to be carefully considered and adjusted, and the resulting formula is highly dependent upon local conditions. For example, both the Kauai sand and gravel had unique properties which had to be compensated for in the mix. *Thus it is not possible to just publish a formula and say, “this is high volume fly ash concrete.” Starting with the basic concept of up to 60% fly ash, water and admixtures have to be adjusted to create a placeable mix that will gain strength at the correct pace* (emphasis added) (Press Release, 1999).

The concrete slab for the Hawaiian temple was completed in September of 1999 and there are no cracks to date. A technical article is currently under review to be published in *Concrete International*.

4.0 Summary and Conclusions

Conclusions that may be drawn from the literature review are presented here. A chronological summary of the information gathered in the literature review is provided in Table 1.

Perhaps the most important finding from this literature review is that cementitious materials used nearly 9,000 years ago have survived intact to the present day. These materials include lime mortars and cements used between 8,800 and 4,000 years ago and concretes used between 3,000 and 800 years ago. Another important finding is that many of the concretes that have survived over such long periods have been some form of pozzolanic concrete. It would, therefore, appear that similar cementitious materials under similar circumstances should be able to last at least as long.

Table 1. Ancient Cementitious Materials

Age	Use	Composition
8850 years	Floor slab in an ancient village in Israel.	Lime cement composed of crushed limestone, ash, and sand.
4500 years	Egyptian pyramid mortar.	Mortar mix composed of gypsum, anahydrite, calcite, argillaceous limestone, and sand.
3000 years	Water tank in Greece.	Concrete mix composed of siliceous gravel, calcareous aggregates.
1800 years	Pantheon. Roman Building.	Concrete mix composed of various aggregates and pozzolanic mortar.
1500 years	Roads in Rome.	Concrete mix composed of small gravel, lime, pozzolanic material.
900 years	Roof slab in Mesoamerica.	Concrete mix composed of calcite, quartz, diopside and nepheline composites.
< 1 year	Temple foundation in Hawaii.	Concrete mix composed of modern fly ash.

The Hawaiian temple example suggests that the blending of ancient and modern concrete technologies may provide a durable long-lasting concrete that could assist the DOE in meeting its goals for marking the WIPP site. The examination of historical uses of concrete suggests that it should be possible to produce a concrete that will survive for a substantial portion of, if not the entire, 10,000-year regulatory period.

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