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Nineteenth Century Concrete in Seguin, Texas: Construction Materials & Techniques

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Nineteenth Century Concrete in Seguin, Texas: Construction Materials & Techniques

by

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Dedication

To my wonderful parents, your personal ambition and strength provide me with continual inspiration. I cannot begin to thank you both for the endless words of encouragement, advice, and much needed positivity. Your support has equipped me with the perseverance to complete this project.

To my brave and beautiful sister, Amy. I find my motivation in your continued professional and academic achievements. Your unwavering desire for greatness never ceases to amaze me. As Mom says, “imitation is the greatest form of flattery.”

With love from your daughter and sister.

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Abstract

Nineteenth Century Concrete in Seguin, Texas: Construction Materials & Techniques

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This investigation centers on early concrete technology used in Seguin, Texas, during the mid-19th century. Over the course of fifty years, more than ninety concrete structures were built in Seguin. Over the last century, these have dwindled to twenty extant structures. Much of the previous Seguin concrete era research has focused on the historical narrative and architectural description. This study aims to answer questions that previous research has not — it investigated the raw materials used in making Seguin's concrete. The results provide new information about the Seguin concrete structures, providing guidance for their long-term maintenance.

The materials analysis uses instrumental techniques such as scanning electron microscopy, energy dispersive x-ray spectroscopy, and x-ray diffraction to determine the chemical composition and crystalline structure of the cement binder from several extant structures in Seguin. Gathering both qualitative and quantitative data for the binder allowed us to identify the raw materials used in the concrete and better understand the construction methods. Studying the materials and methods increased our understanding of these historic structures and will inform future preservation efforts.

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Chapter I: Introduction

This thesis investigates early concrete technology used in Seguin, Texas. Texas vernacular architecture of the late 18th to mid-19th century employed traditions of the people who inhabited the territory. Early Texans used concrete technology based on their geographic location. The concrete in Seguin pre-dates the availability of Portland cement in the United States and was not a typical construction material in Texas. Several factors influenced construction methodology and architectural style including climate, economics, and material availability. Between 1840 and 1890, over ninety structures were constructed of concrete. Although the Concrete Era in Seguin has been well documented by local historians since the early 20th century, little of the research has involved material investigation.

Seguin has been called "The Mother of Concrete Cities," due to several factors. The first can be found in Frederick Law Olmsted's travel journal of his time in Texas. As he traveled along the Guadalupe River in 1854, Olmsted visited Seguin and documented his impression of the many buildings made of concrete.[1] The same year, the *Texan Mercury* published an article using the nickname, highlighting the city's use of local material to construct the buildings.[2] The leading proponent of concrete construction in Seguin was also the man to whom the technology is attributed: Dr. John E. Park. Park holds several patents for the material and its manufacture.[3]

Seguin has records of its concrete structures because of documentation efforts starting in the early 20th century. A few of the buildings were documented through the

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[1] Frederick Law Olmsted, *A Journey Through Texas* (Austin, TX: University of Texas Press, 2010): 231.
[2] "Sebastopol," *National Register of Historic Places Nomination*. Files of Texas Parks and Wildlife (Austin, Texas, 1970), 35-36.
[3] John E. Park, 1871. *Improvement in the Manufacture of Cement,* US Patent 138,924, filed April 6, 1871 and issued May 13, 1873.

Historic American Building Survey in the 1930s. During the 1970s, National Register nominations were completed for several individual structures and for the Seguin Historic Commercial District; the district includes many extant concrete structures. [4]

Many amateur historians wrote about the history of Seguin and Guadalupe County. Two of the most prominent of these authors were Willie Mae Weinert and Father L. J. FitzSimon. FitzSimon's book was undertaken as part of the city's centennial celebration in 1938.[5] A drawback to this publication is that references were not cited. Father FitzSimon compiled the available primary documents to describe the events surrounding the city's establishment. He provides detailed accounts of Seguin's founding and also answers some of the questions about the city's concrete construction. FitzSimon suggests that the raw materials for the cement binder were quarried locally; this differs from Weinert, who uses the term "limecrete" and suggests that the raw materials were quarried in San Marcos. [6] Both historians attributed the concrete formula to Dr. Park, even though they differed in opinion regarding the source of raw materials.

Willie Mae Weinert was an amateur historian who collected material from the Texas State Library Archives, the University of Texas Archives, county records, and newspapers in order to piece together the history of Guadalupe County. Her research of Seguin's first 100 years was intended as two volumes, however, only the first was published. In cases where she could not find evidence, Weinert had to rely on oral history and traditions. Weinert's book is one of the first publications to use the term "limecrete"

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[4] Additionally, a historic resource survey was completed during the 1970-1980s that documented all historic resources in downtown Seguin.

[5] L.J. Fitzsimon, Seguin, Texas (Seguin: City of Seguin, 1938).

[6] Willie Mae Weinert, *An Authentic History of Guadalupe County* (Seguin: The Seguin Enterprise, 1951).

to describe concrete construction in Seguin.[7] Limecrete is a technical term that we will return to and define later.

In 1980, Vincent Paul Hauser, a graduate student in the University of Texas at Austin School of Architecture, wrote his thesis on "A Survey of the Technologies Contributing to the Concrete Era of Seguin, Texas in the Mid-Nineteenth Century."[8] Hauser described the development of early concrete technologies, such as cob, pise, tabby, and shellcrete. Like his predecessors, Hauser attributes Dr. Park with creating the formula for concrete in Seguin. He acknowledges the similarities and relationship that Park's concrete shares with these earlier building methods. While Hauser does spend time discussing natural cement, "gravel wall," and limecrete technology, his thesis is best known for its documentation of then-extant concrete structures in Seguin.

While much research has been conducted on the concrete construction methods, historical narrative and architectural description, a materials analysis of the concrete will provide new insight to the concrete era in Seguin.

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[7] Willie Mae Weinert, *An Authentic History of Guadalupe County* (Seguin: The Seguin Enterprise, 1951), 59.

[8] Vincent Paul Hauser, "A Survey of the Technologies Contributing to the Concrete Era of Seguin, Texas in the mid-Nineteenth Century," Master of Architecture thesis (The University of Texas at Austin, 1980).

Chapter II: Seguin: *Mother of Concrete Cities*

In 1838, just two years after the war for Texas Independence, the present city of Seguin was founded as Walnut Springs. By 1839, many of the settlers disliked the name and voted to change it to Seguin, honoring Juan N. Seguin for his service during the Texas Revolution. Seguin is situated on the Guadalupe River approximately thirty miles northeast of San Antonio. The Concrete Era (c.1840-1890) occurred simultaneously with wood frame, brick, and masonry construction. Concrete construction included residences, commercial and municipal buildings, as well as cisterns and cemetery walls. At the height of the era, there were over ninety concrete structures. This was also a time of marked increases in population and wealth, evidence of which was seen in the architectural style and rate of new construction.[9] It is because of the prominence of concrete construction that a reporter from the *Texan Mercury* proclaimed Seguin "The Mother of Concrete Cities."[10]

CONCRETE CONSTRUCTION IN SEGUIN

Over the last century, many concrete structures have been lost, leaving a little over twenty extant structures. Most concrete structures can be identified by their thick walls and pebbled surfaces. Some of the structures, however, have only a wall or section of the original material or are obscured by slipcovers, making these structures difficult to detect. In 1854, Frederick Law Olmsted traveled through Texas and wrote a journal recounting his expedition in great detail. Olmsted made his way along the Old

[9] Editorial, "The Mercury Correspondance," *Texan Mercury*, December 10, 1853.
[10] Editorial, "Names of Cities," *Texan Mercury*, June 10, 1854.

San Antonio Road from Louisiana to San Antonio, Texas and stopped in Seguin during the height of its concrete construction. He recorded his first impressions of Seguin, naming it “the prettiest town in Texas” of all the ones he had visited. [11] He continued to report on the natural landscape, the abundance of supplies, and his pleasure with the accommodations. When describing the buildings he encountered, Olmsted wrote:

> A number of buildings in Seguin are made of concrete–thick walls of gravel and lime, raised a foot at a time, between boards, which hold the mass in place until it is solidified. As the materials are dug from the cellar, it is a very cheap mode of construction, is neat in appearance, and is said to be as durable, while protected by a good roof, as stone or brick. One man may erect a house in this way, calling in mechanics only to roof and finish.[12]

Remaining physical evidence corroborates Olmsted’s description of the construction method with only one exception—many of the remaining structures do not have cellars from which the material would have been dug. Physical evidence does indicate that the structures were poured in place in one-foot lifts as Olmsted described. In his observations he did not specify the wall thicknesses, but most of the walls are fifteen inches. When the concrete is exposed, either from interior or exterior, in areas no longer covered in stucco, characteristic pour lines are visible.

Originally, these structures would have been covered by stucco to protect the concrete—stucco fragments remain on some structures. Today, the concrete structures have either smooth or pebbled non-original stucco, but it is indicated in early photographs that the original finishes were either smooth or scored to simulate mortar joints. As the

[11] Frederick Law Olmsted, *A Journey through Texas* (Austin, TX: University of Texas Press, 2010), 231.
[12] Olmsted, 231.

stucco weathered, the concrete was left unprotected, causing binder erosion. This gave the buildings a pebbled texture, which may have been interpreted as the intended finish during stucco replacement. The original stucco was most likely composed of lime, sand, and water. During repair campaigns, the coatings have been renewed with a variety of cementitious mixes. In most cases, the stucco appears to be Portland cement. This type of stucco is incompatible with Seguin concrete because it is too hard and impermeable.

The concrete itself is composed of binder, coarse aggregate, and sand. The large aggregate, unless (purposefully) concealed with smooth stucco, gives the concrete a pebbled appearance. Originally, the concrete would have had a flat surface due to use of board forms during construction, however, binder erosion has exposed the large aggregate and given it a new surface texture. The board forms would have been reused throughout construction, but the spreaders used to across the forms would have been left within the wall. The spreaders were rough-cut wood with varying thicknesses. In some places, spreaders are visible in the walls.

In recent decades, many speculate that the raw materials for Seguin concrete may have been quarried in San Marcos.[13] In 1854, the *Texan Mercury* published an article titled "Where Does it Come From?" in which the editor stated:

> Where does it come from?— Those of our readers who have seen our town, know that many of our buildings are concrete. The gravel and lime is (*sic*) procured within the corporation limits, thus making "holes in the ground;" and these are left

[13] Information provided during interviews with professionals who worked in Seguin. Killis Almond, phone conversation with author, November 26, 2013. Vincent Paul Hauser, interview with author, October 17, 2013.

> to catch all they can, as though someone had "digged (*sic*) a pit for his adversary."[14]

The author continues, noting the dangers of open pits within the city limits because three people fell into one of the excavations at night time. This article fails to mention an exact location for the source of lime and gravel, but confirms that the material was locally sourced.

In May of the same year, the *Texan Mercury* reported that a new contract had been signed for constructing a jail. It stated that the building would be located in the eastern part of Seguin, near the hole where citizens procured gravel for making concrete, specifying that it is the same hole in which the three people fell.[15] On the 1885 Seguin Sanborn map, lot 26 notes that the one story stone jail is located 340 feet east of the Magnolia Hotel.[16] This indicates that the jail was constructed on either lot 27 or lot 28. Lot 27 is partially included on the 1902 Sanborn map, with a note that there is 155 feet to the two story brick county jail. It was not until 1906 that the structure is depicted on the Sanborn map; it is located on the northeast corner of lot 27 and is shown as a two story brick structure with slate roofing. Now lot 27 can be located by its relationship with the Magnolia Hotel. By studying the progression of lot 26 and 27 on the Sanborn maps, we hoped to gain information about the location of the reported excavation site. On-site investigations in this area did not prove fruitful, as much of the site has been developed. The northeast corner now has an asphalt parking lot and a three story structure.

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[14] Editorial, "Where Does it Come From?," *Texan Mercury*, February 11, 1854.
[15] *Texan Mercury*, May 20, 1854.
[16] See Appendix B for a copy of each of the historic Sanborn maps.

During the progress of this research, I was contacted by the Seguin Chapter of Master Naturalists. As part of the Seguin Parks and Recreation *Parks Master Plan*, the group is investigating the city-owned Hoerman property to identify native and invasive vegetation.[17] The northeastern part of the property has a historic structure that has been previously identified as the Moses Campbell house.[18] During their investigations in the wooded areas, they found a small clearing that contained a large pit. The approximate location of the pit is less than 0.1 mile from the house on the property; it is also about 0.25 miles from Sebastopol. The pit is approximately 15x20 feet with an irregular shape. Over time, debris and vegetation have filled the pit, leaving its true depth to speculation. At its deepest point, it was about two feet below ground level. Weathered limestone forms the edges of the pit; without much effort, one could obtain a hand sample.

According to the on-site investigation, the pit appears to have been man-made. Given its proximity to concrete structures, the pit could very well be a source of raw materials for the concrete construction. Samples were collected from the site and analyzed with x-ray diffraction. The results of this analysis are described in Chapter 4.

PATENTED FORMULA

Another area of contention over the location of the raw materials was a 20th century theory of the origin for this mode of concrete construction. Without other Texas

[17] The Hoerman property is bounded by Vaughn St. to the west, San Antonio St. to the north, Williams St. to the east (with houses adjacent to the property), and Court St. to the south. The southern boundary has been subdivided and is no longer part of the Hoerman property.

[18] This structure is reportedly composed of concrete; however, the house appears to have some walls built of caliche block and other walls of what appears to be pre-Portland cement concrete. The house is boarded up and we were unable to go inside during our site visits. We recommended that the city proceed with an investigation to determine the construction methods and historic significance of the structure.

examples of early concrete construction, many historians have produced conflicting theories from the remaining historic resources. The most prominent and persistent theory attributes the promotion of concrete construction in Seguin to Dr. John Esten Park (1814-1872). Park brought his family from Eatonton, Georgia to Seguin in c.1846. Educated as a physician, Dr. Park has been said to be a self-promoting inventor and chemist. According to Vincent Hauser's 1980 thesis, Park's house was constructed in 1848 and appeared to be "a virtual experimental laboratory," incorporating concrete of various formulas; his home has since been demolished.[19]

In 1848, an association of Seguin citizens chartered the creation of an educational facility for the city. They contracted Dr. Park to erect a two-story concrete building; this building was constructed within a year at an estimated cost of $5,000.[20] The school thrived and became known as the Male Academy. The structure still stands, now known as the St. James Catholic School, and is claimed to be the oldest building in Texas to be in continuous use as a school. The Female Academy was constructed two years later, also of concrete but was destroyed by fire before the turn of the century. In addition to his contract to construct the Male Academy, it is reported that the Guadalupe County Court appointed Park along with other agents to collect donations in money and property for the construction of the new jail in 1854.[21] This jail was constructed near the lime and gravel excavation site; however, it was reported that the jail was contracted to be built by

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[19] Vincent Paul Hauser," A Survey of the Technologies Contributing to the Concrete Era of Seguin, Texas in the mid-Nineteenth Century" (master's thesis, The University of Texas at Austin, May 1980) 96-97.
[20] L.J. Fitzsimon, Seguin, Texas (Seguin: City of Seguin, 1938), 25.
[21] Willie Mae Weinert, *An Authentic History of Guadalupe County* (Seguin: The Seguin Enterprise, 1951) 11.

"Reverend A. Herron, for $2,950.00...to be built of live-oak blocks, two stories high."[22] Other than the Male Academy and possibly his own residence, Park's direct association with Seguin's concrete construction is largely based on oral history.

According to United States Census data, Park and his family remained in Seguin until the Civil War; by 1870 they were located in Warren County, Tennessee.[23] While in Texas, Park claimed the value of his real estate as $50.00 in 1850. In 1860 he claimed the value of his real estate to be $1,500 and his personal estate to be $1,000.[24] This could be attributed to the receipt of a land grant in April 1858 for 640 acres.[25] Not much is known about Park's final years; he moved to Austin, Texas, sometime between 1870 and 1872, where he died of heart disease. Prior to his death, Park applied for a number of patents, all of which were issued posthumously. [26]

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[22] *Texan Mercury*, May 20, 1854.

[23] United States Census Bureau, *1860 Census Data*, Seguin, Texas, Guadalupe County, 6. United States Census Bureau, *1870 Census Data*, McMinnville, Tennessee, Warren County, 8.

[24] United States Census Bureau, *1850 Census Data*, Seguin, Texas, Guadalupe County. United States Census Bureau, *1860 Census Data*, Seguin, Texas, Guadalupe County, 6.

[25] Texas Land Office, Court of Claims 006327, April 16, 1858.

[26] The first patent was for the "Improvement in the Manufacture of Artificial Stone." This patent specified that material should be used from the calcareous beds of Western (now central) Texas where the clay content is not 25% of the entirety and to add clay until 25-30%. The mass is then to be moistened by salt-water and then molded into desired shape and size, then placed to dry (similarly to brick). The 'bricks' were to then be fired in the specified kiln at a high heat for four hours to develop incipient cementation. In effect, this patent describes a process for making concrete masonry units (CMU). Although CMU construction has not been found in Seguin, there is a likelihood that Park could have experimented with this construction at his home prior to patenting the work. John E. Park, Improvement in the Manufacture of Artificial Stone, US Patent 127,264, dated May 28, 1872. See Appendix A. A second patent was specified for the "Improvement in Cement-Kilns." This specification included accompanying drawings that showed Park's improved arrangement of the furnace-flues. He made these improvements to ensure that the cement material would be heated equally no matter its placement within the kiln. In previous kiln designs, one side of the kiln was subjected to a heat that was more intense than the other based on the placement of the flues and hearths; the resulting effect would impair that quality of the cementitous material. This kiln was specified to be used for the manufacture of artificial stone. John E. Park, Improvement in Cement-Kilns, US Patent 133,664, dated December 3, 1872. See Appendix A.

Each patent was submitted by John E. Park of La Vergne, Tennessee. As previously mentioned, Park registered in McMinnville, Tennessee (approximately sixty miles Southeast of La Vergne) during the 1870 U.S. Census. Although the difference in Park's locations could be attributed to travel or to the potential acquisition of post-Civil War land grants in Tennessee, these theories are both speculative without accompanying historic documentation.

The most relevant patent was issued in 1873, but the application was sent in 1871. This is the only patent issued to Park that specifies the original date of application. Park, or someone on his behalf, had begun the patent process prior to his death in 1872. This patent is for the "Improvement in the Manufacture of Cement." Park specifies that his invention relates to new hydraulic cement. He states that the cement is composed of "any of the forms of lime, with thirty to forty percent of clay (alumina and silica), five to ten of fine sand (silex), and five percent of soda (carbonate, muriate, or caustic) or potash."[27] The materials are then to be mixed together; the amount of mixing is determined by how much clay is added. Park closes by stating that the proportions of the materials may vary somewhat, but the preferred proportions will produce a cement that will not set in water, but will set in air and the hardening process will continue in water. This is an apparent contradiction because hydraulic cement sets under water and Park calls his patented material hydraulic cement, but says that it does not set under water.[28]

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[27] John E. Park, 1871. *Improvement in the Manufacture of Cement*, US Patent 138,924, filed April 6, 1871 and issued May 13, 1873. See Appendix A.

[28] Hydraulic cement will be discussed in greater detail in Chapter III, " Concrete Technology".

When considering the extent of Park's contribution to the concrete era of Seguin, his documented activities and patents must be weighed against the oral histories that circulate among the city's history. Park was clearly in Seguin during the concrete era, but there are only two documented instances of his participation in the construction of buildings, only one of which was concrete. The physical evidence of his residence has been lost, as have many of the potential primary sources that could have tied him more closely to the concrete construction. Something must be said, however, for the oral traditions that speak to Park's connection with Seguin's concrete construction. Oral histories are valuable resources for smaller towns that do not have a physical repository for archival documents. At the local level, the community has the Seguin-Guadalupe County Heritage Museum, but its collections are predominantly photographic materials that date from the 20th century.

The stories connecting Park to concrete began to be recorded in 1938, during Seguin's Centennial Celebration. Local historian, Willie Mae Weinert, wrote a book titled *An Authentic History of Guadalupe County* in which she had to rely on many local oral traditions and histories for information of undocumented events.[29] These stories should not be discounted, but should be instead compared to existing documents to support their claims.

Park's patents serve as an additional indication of his interest and activity in the manufacture of cement. The cement described in Park's patent is manufactured hydraulic

[29] Willie Mae Weinert, *An Authentic History of Guadalupe County* (Seguin: The Seguin Enterprise, 1951).

cement, whose formula could have been used for the construction of the Seguin concrete buildings. A materials analysis is necessary to determine whether Park's mix was in fact used for the construction of the Seguin buildings. This will be discussed in detail in a later chapter. As mentioned earlier in this chapter, the concrete structures of Seguin have dwindled from more than ninety to about twenty. For this study, five of the remaining structures were selected for materials analysis. They will be described in the subsequent pages.

EXTANT STRUCTURES INCLUDED IN THE STUDY

The structures included in the study were selected according to the following criteria. The first criterion was that permission had to be easily obtained from the owner to extract samples from structures. The second criterion was that the original material was accessible. The final criterion was that the construction date was within the period of significance (c. 1850-1880) and that the structure was in reasonably good condition. As is the case in many historic buildings, access to original materials is of primary concern. The concrete structures of Seguin have often been renovated or encapsulated with 20th century stucco, making it difficult to access original materials. The structures that fit the criteria are listed below with a brief description.

Magnolia Hotel, 1847

Figure 2.1: Magnolia Hotel, Author's image, September 12, 2013.

The Magnolia Hotel was originally constructed in 1838 by James Campbell as a modest log structure, but was sold in 1845 to Joseph Johnson who is said to have constructed the concrete addition. It was during this time that the structure was operated as a hotel and no longer as a residence. Sometime after the concrete portion of the hotel was completed, a two-story wood frame addition was constructed on the northern part of the lot. Over the next thirty years, the hotel changed ownership multiple times and no longer operated as a hotel in the early 1900s. After ten years of vacancy, the structure was re-opened as a boarding house, but was not listed as the Magnolia Hotel. The 1885 Sanborn shows it on block 26.[30] The concrete portion of the hotel is rectangular in form and is the southernmost part of the building.

[30] See Appendix B for map.

The concrete structure has eight inch thick walls.[31] The floor plan has been remodeled and is no longer indicative of the hotel plan. It has a pitched roof with wide overhang that protects the concrete walls. The exterior is now covered in smooth white stucco. The original material is visible from the interior in a narrow passageway from the concrete structure to the wood framed structure where a small area of exposed material is located by the window in the hallway.

Riverview Cemetery Walls, c.1850

Figure 2.2: Riverview Cemetery Wall, Author's image, September 19, 2013.

Not much is known of the Riverview Cemetery perimeter walls. It appears that they once surrounded the cemetery, most likely offering protection from animals that might have disturbed the graves. The cemetery was too far from downtown to be included in the Sanborn maps, but was said to have been constructed in the 1850s.[32] Only small sections of the cemetery walls still exist; most of them have fallen. The walls

[31] Most of the other structures in this study have wall thicknesses of 15 inches. The Magnolia Hotel is one of the oldest extant concrete structures in Seguin. Since it is such an early example, it could account for the variance in wall thickness.

[32] See Appendix B for maps.

are approximately four feet tall and have an average thickness of twelve inches. Much of the binder has eroded exposing large aggregate and any evidence of a previously applied stucco is not present.

Sebastopol, 1854

Figure 2.3: Sebastopol, Author's image, September 12, 2013.

Sebastopol was constructed in 1854 and is the best known of the concrete structures in Seguin. This can be attributed in large part to its publicized restoration by Texas Parks and Recreation in the 1970s-1980. The structure itself was a residence from 1854 to the 1960s, and changed ownership several times. Its construction and design are attributed to German immigrant, Tobias Meineger. The most famous owner and resident was Seguin's mayor, Joseph Zorn. The structure was documented with measured drawings in the 1930s as part of the Historic American Building Survey and the first steps towards its preservation were through the advocacy of the Seguin Conservation Society in the 1960s.

The house has a raised plan with a basement accessible from three sides. It is considered to be in Greek revival style, with a hipped roof hidden behind its parapet walls. The roof is reported to function as a water collection and cooling system by storing water on the roof and diverting some of it to a basement cistern. The exterior walls are covered in smooth white stucco, and the rooms upstairs are plastered. The only access points to original concrete materials are found in the basement interpretive areas. From here, the concrete walls are exposed and pour lines are visible. The walls are approximately fifteen inches in thickness and were poured in one foot lifts. It must be noted that some of the exterior facing walls were deteriorated to such a point that they were demolished and reconstructed. Samples were extracted from the basement spaces from areas that were representative of original material.

Moses Campbell Commercial Building, c.1856

Figure 2.4: Moses Campbell Commercial Building, Author's image, May 1, 2014. The photo was taken from the southeast corner of the E. Court St. and N. Austin St. intersection. The Moses Campbell Commercial Building is the corner structure. The adjoining buildings to the west and to the north were renovated in the mid to late 20th century to join the Moses Campbell building. The concrete wall is located between the Campbell building and the building to the north.

Figure 2.5: Moses Campbell Commercial Building, Author's image, September 19, 2013.

The Moses Campbell Commercial Building was constructed for Moses Campbell in 1853-1856. Located on the northwest corner of Austin and Court Streets, the second story served as the offices for the *Seguin Journal*, a weekly newspaper. In the 1880s the structure was remodeled and much of its concrete material was replaced with brick from the factory of August Dietz. At this time the building served as the law office for John Ireland. In the 1885 Sanborn map, the building is shown on the southeast corner of block 36 and has only one concrete wall depicted. A renovation of the building in the early 2000s exposed the concrete wall, which has been left exposed. The building is currently vacant.

This wall is fifteen inches in thickness, but differs from the other buildings in this investigation as it has large caliche blocks at the bottom three feet of the wall. The pour lines are visible on the concrete portion. Wood spreaders from the original formwork are still embedded in the wall and are only seen due to the incidental wall exposure. If demolition was not attempted, we would not be able to see the concrete wall in cross-section and the construction information might have remained hidden during the lifespan of the building. The wall had 20th century sheetrock applied to both sides, but was presumably covered in a period plaster when first constructed.[33]

Old Baptist Church, c. 1878

Figure 2.6: Old Baptist Church, Author's image, September 12, 2013.

The Old Baptist Church was constructed on block 23 from 1876-1878 by the Baptist congregation. The building operated in a religious capacity from its construction to c. 1960. From then until 2005 the building was used as a print shop; after this time the building served a dual purpose of apartment house on the south end and a food bank on the north end. The original structure was rectangular in plan, as can be seen in the 1885 Sanborn map.[34] Around the turn of the century, a small wooden addition was made on the northwest side of the building. The last renovation, completed c.1900-1910, included a large addition connected to the south end of the original structure. The addition was a wood framed structure covered in cement stucco to appear similar to the original concrete portion of the building.

[33] When the wall was discovered, the construction workers had demolished the center of the load bearing concrete wall and it had to be stabilized with a steel column and beam. Construction was halted because of lack of adequate funds to complete the project.
[34] See Appendix B for maps.

The original structure has walls approximately fifteen inches in thickness. The exterior of the building is covered in pebble finish replacement stucco that imitates the appearance and character of the original concrete material after the binder has eroded. Sections of the stucco have become detached, exposing the original concrete and making the pour lines easily visible. The pour lines appear to be consistent with one foot lifts, but are only exposed in small sections.

Chapter III: Concrete Technology

To understand the concrete era in Seguin, one must have a basic knowledge of concrete mixes and cement binders. For the purposes of this research, I will only discuss the types of concrete that were prevalent in Texas during the 19th century. The following descriptions are not meant to be exhaustive, but will instead provide a brief depiction of the concrete technology in Texas during the concrete era in Seguin. In its most basic terms, concrete is essentially a mixture of binder, aggregate, and water.[35] There are variations of this formula that contribute to the compressive strength and workability of the material. Lime, in a variety of forms and sometimes with additives, was arguably the most widely used ingredient in 19th century concrete. The composition of limestone, the raw material for lime, determines the type of cement binder that can be produced. Prior to the industrialization of cement manufacturing, limestone was crushed, burned in kilns, and hydrated with water to make lime. Industrialization allowed for more precise mix proportions, but used a similar process.

TYPES

During the 19th century, cement binders were most likely made of lime (limestone that has been crushed and burned). Some of the binders found in concrete and mortar during the 19th century are:[36]

1. High Calcium Lime (limestone with few impurities)

[35] Jerry Ingham, *Geomaterials Under the Microscope: A Colour Guide* (London: Manson, 2011),75.
[36] Harley J. McKee, *Introduction to Early American Masonry, Stone, Brick, Mortar, and Plaster* (Washington, D.C.: National Trust for Historic Preservation, 1973)

2. Hydraulic Lime (limestone with 20-30% clay, that allows it to set in water and adds strength)
3. Natural Cement (Cement "rock" with 40-60% clay)
4. Portland Cement (limestone with clay or shale with definite proportions)

The concrete in Seguin was made before Portland cement became available in the United States c. 1870. Over the last seventy years, the concrete has been locally described in two ways, simultaneously; however, each description indicates the use of a different binder. The first description is "limecrete"; this terminology indicates the use of a high calcium carbonate lime binder. The second description is Dr. John E. Park's Concrete; this references the patent the Dr. John E. Park holds for the "Improvement in the Manufacture of Cement."[37] Park's patent is for a new hydraulic cement binder using lime with 30-40% clay. The two descriptions, although used interchangeably, describe two different types of materials that behave in different ways. An additional theory is that the citizens of Seguin used limestone quarried locally for the manufacture of lime. If this was the case, the composition of the binder material would correspond to the composition of the local limestone deposits.

Limestone deposits are calcium carbonate, but can contain impurities such as magnesium, silica, alumina, iron oxide, sulphur, or alkalis.[38] In the Seguin area, the deposits are from the Quaternary period and are considered to be too recent to be of

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[37] John E. Park, 1871. *Improvement in the Manufacture of Cement*, US Patent 138,924, filed April 6, 1871 and issued May 13, 1873.
[38]Edwin C. Eckel, Cements, Limes, and Plasters: Their Materials, Manufacture and Properties (New York: Wiley, 1909), 94.

interest for a geological investigation; however, the Seguin area has mostly fluviatile stream deposits, meaning that the composition has gravels, sands, a high percentage of calcium carbonate from caliche and more than likely has clay impurities.[39] The clays would most likely be kaolinite (aluminum silicate) and the limestone would also have trace amounts of quartz. Caliche is a sedimentary rock where calcium carbonate binds other materials together; such as gravel sand, clay, and silt. It is found near the ground surface and can be easily quarried in blocks for building construction. Caliche has also been used as a raw material for lime production when it is relatively pure calcium carbonate. Because of its proximity to the Guadalupe River, Seguin has a high concentration of alluvial deposits; these deposits most likely provide the coarse aggregate used in 19th century concrete structures.

Shellcrete

Similarly to the construction method of Tabby that is found in coastal areas of the southeast, Shellcrete uses oyster shells, found locally, as the large aggregate in the concrete mixture. The binder in shellcrete is also made from oyster shells or is quarried from local limestone deposits. Shellcrete binder typically has impurities of silica, iron oxide, and alumina. In Texas, Shellcrete is found near the gulf coast. The best example of shellcrete construction is Fulton Mansion in Rockport, Texas. Built from 1874-1877, the mansion was constructed contemporaneous to the Old Baptist Church.

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[39] Earle Mcbride, Interview with author, February 21, 2014. Jim Dyess, Phone conversation with author, April 3, 2014.

Limecrete

Limecrete is a concrete mixture that has large aggregate and uses relatively pure limestone for the source of lime. From available research, it appears that the only reported "limecrete" buildings in Texas are located in Seguin. The Texas Historical Commission, Texas Parks and Wildlife, and Vincent Hauser's thesis associate Dr. John E. Park with the formula for limecrete. As discussed in previous chapters, Dr. Park's patented formula is for hydraulic cement and not for limecrete, as it has been described. The use of the term limecrete to describe the Seguin era concrete has led to assumptions about the composition of the concrete. This could lead to the selection of inappropriate repair materials.

The Players

One of the earliest questions guiding this project was surrounding Dr. John E. Park and his knowledge about concrete construction. Up until this point we have discussed Park in isolation and have not considered his contemporaries. During Park's lifetime (1814-1872), two other men were proponents of early concrete construction: phrenologist and lecturer, Orson Squire Fowler (1809-1887), and builder Joseph Goodrich (1800-1867). The better known of the two was Fowler, who wrote *The Octagon House: A Home for All* originally published in 1848. Fowler resided in New York and New Jersey and was exposured to a variety of people and technological advancements. Within this publication Fowler called for people to construct buildings of "gravel wall." He describes in his book:

> [gravel wall] is made wholly out of lime and stones, sand included…And pray what is lime but stone? Made from stone, the burning, by expelling its carbonic

> acid gas, separates its particles, which, slacked and mixed with sand and stone, coats them, and adheres both to them and to itself, and, reabsorbing its carbonic acid gas, again returns to stone, becoming more and more solid with age.[40]

During lectures, Fowler promoted the benefits of gravel wall technology and the octagonal house. Additionally, Fowler would travel to see concrete buildings in other parts of the U.S. He wrote about his visit to Milton, Wisconsin, to see Joseph Goodrich.

Goodrich, originally from Massachusetts, moved to Wisconsin searching for opportunity in westward lands. Upon his arrival in Wisconsin, Goodrich built several buildings of concrete. His personal residence, the Milton House, was constructed in 1845 around the same time that the Magnolia Hotel (1847) was constructed in Seguin. His home was octagonal and used lime binder with course gravel and sand. Fowler visited Goodrich in 1850, and in a new addition of his book, stated that Goodrich developed this mode of construction. Fowler never makes mention of Park in any of his editions, but his travels were predominantly in the northern states of the U.S. It is also curious that in 1854, during Fowler's lifetime, Frederick Law Olmsted traveled through Seguin and wrote about the concrete construction. Fowler might have had knowledge of the structures through Olmsted's published travel journal.

It is difficult to prove that Fowler had knowledge of Park and the concrete structures in Seguin, but one would assume that he would have written about either if he had knowledge of their existence. It is also difficult to prove whether Park knew about Goodrich and Fowler. The most important thing to note is that similar concrete technology was being developed in the U.S. at the same as Seguin's Concrete Era.

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[40] Orson Fowler, 18-19.

Chapter IV: Materials Investigation

The concrete structures in Seguin, Texas, were constructed over a fifty-year period prior to the availability of Portland cement in Texas. On-site investigations provided physical evidence that each poured-in-place structure was constructed in one-foot lifts with thick walls. As mentioned in Chapter III, the Seguin concrete construction was happening contemporaneously to the shellcrete construction on the coast on Texas. In both cases, the structures were poured in place with thick walls. We can understand much about how these concrete structures were constructed based on physical evidence; however, identifying the materials used is more complicated. This chapter discusses the analytical techniques used to identify the composition of the binder used in Seguin concrete.

Structures

As mentioned above this study examined five structures. These structures were in reasonably good condition, had accessible original material, and were constructed during the period of significance (c. 1850-1880). The samples were collected on two days in September 2013 from interior and exterior locations. Although the samples were extracted from areas with different weather exposures, they were all friable and crumbled upon extraction. Due to this condition, varying amounts of aggregate were collected with the powdered binder material. The structures are described in detail in Chapter II. The following section includes information that is specific to sample collection.

Magnolia Hotel, 1847

Figure 4.1: Magnolia Hotel, Author's image, September 12, 2013.

Figure 4.2: Window frame & exposed concrete, Author's image, September 12, 2013. The photo shows the small exposed area adjacent to the window frame that allowed for sample extraction.

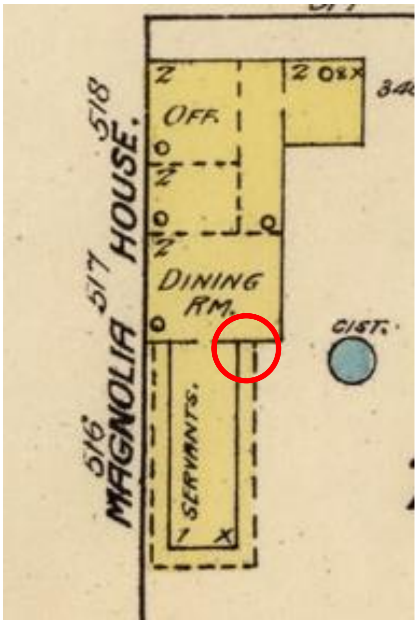


Figure 4.3: Lot 26, 1885 Sanborn Map, small section showing the northwest corner of block 26; circle shows the area where the sample was taken. The map portion indicates woodframe construction by using yellow; however, the southern portion of the building is concrete, which is typically indicated by blue on the Seguin Sanborns—some versions indicate this structure in blue.

The Magnolia Hotel was constructed in two phases: the first structure is to the north and is woodframe construction; the second is to the south and is constructed of Seguin concrete. The graphic shows the basic footprint of the structure as it stands today. Due to recently completed renovations, the exterior and most of the interior of the structure have been covered in stucco with no exposed areas; however, the small hallway connecting the woodframed and concrete portions of the building has an area of exposed original concrete material. A small board-covered window has an exposed area of approximately 15”x 5” at its greatest height and width, respectively.

Riverside Cemetery Walls, c.1850

Figure 4.4: Riverside Cemetery Wall, Author's image, September 19, 2013.

Figure 4.5: Riverside Cemetery Wall, Author's image, September 19, 2013. This image shows the areas where two wall sections are joined. Samples were extracted just to the outside of this joint.

The Riverside Cemetery walls once surrounded the cemetery, but now only exist in isolated sections. The cemetery walls were most likely constructed in sections that were connected with a mortar-like material. Even though some sections of the cemetery walls stand in their original locations, others lie fallen in single sections. The standing walls are approximately four feet high, with an average thickness of fifteen inches. Stucco loss and binder erosion have exposed large aggregate that projects from the wall surface. The upper two feet of the walls have evidence of biological growth. Samples were extracted from the area closest to the expansion joint on the outer portion of the wall and were taken from both the upper and lower portion of the wall in the described area.

Sebastopol, 1854

Figure 4.6: Sebastopol, Author's image, September 12, 2013.

Sebastopol has a raised plan with a basement that is accessible from three sides. During the 1980s restoration the exterior of the structure was restuccoed, but portions of the interior of the basement were left exposed for interpretive purposes. In this space, full heights of the concrete walls are left exposed making the pour lines easily visible. The walls are approximately fifteen inches in thickness and were poured in one foot lifts. Some of the exterior walls were deteriorated to such a point that they were demolished and reconstructed with in-kind materials.

Moses Campbell Commercial Building, c.1856

Figure 4.7: Moses Campbell Commercial Building, Author's image, September 19, 2013.

Figure 4.8: Wall section, Moses Campbell Commercial Building, Author's image, September 19, 2013.

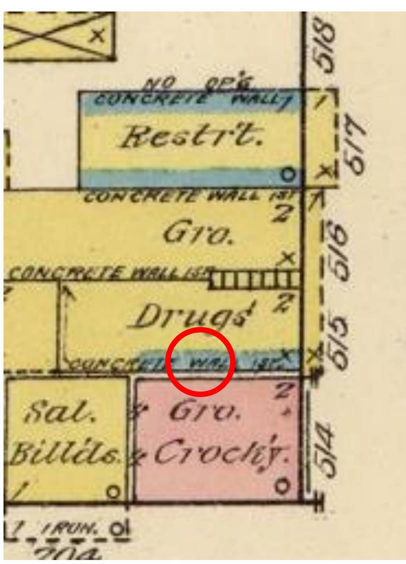


Figure 4.9: 1885 Sanborn map section, showing the southeast corner of lot 36. The east bounding road is N. Austin Street and the south bounding road is Court Street. The red circle indicates the area where the sample was extracted. The concrete wall is located between the red structure (brick construction) and the yellow structures (wood-framed).

Most of the structure that is known as the Moses Campbell Commercial Building is constructed of brick; however, a small portion of original concrete material survives. There is one concrete wall within the structure located toward the southeast corner of the building. This wall was re-discovered during a renovation of the building in the early 2000s and has been left exposed during its vacancy. The wall is fifteen inches in thickness, with the pour lines easily visible. Wood spreaders from the original formwork are still embedded in the wall and are only seen due to the incidental wall exposure. Although this wall has combined caliche block and concrete construction, it is included because it shows an alternative method used in Seguin concrete construction. This wall is the only structure where concrete is seen in cross section.

Old Baptist Church, c. 1878

Figure 4.10: Old Baptist Church, Author's image, September 12, 2013.

The Old Baptist Church's original concrete structure was a rectangular plan, but has a later wood-framed addition on the northwest side of the building. The addition is a wood framed structure covered in stucco that appears similar to that of the original concrete portion of the building. The original structure has walls that are approximately fifteen inches in thickness. The exterior of the building is covered in pebbled non-original stucco that imitates the appearance and character of the concrete material underneath. Sections of the stucco are detached, exposing the original concrete and pour lines. The pour lines appear to be consistent with one foot lifts, but are only visible in small exposed areas. Samples were extracted from within an interior wall in the entranceway and from roof access on the second story where stucco was detached.

PRELIMINARY EXAMINATION

Several samples were extracted from each structure. One of the difficulties in extraction was to keep the samples intact during removal and transportation. The binder material is friable and powders with little to no contact. The preliminary investigation uses visual observation and color matching to identify representative samples for analysis. The visual observations confirm that all of the samples have a similar binder color with only small differences in the detectable value. Samples extracted from exposed exterior locations have more soiling. Overall, the distribution of fine aggregate and the binder color appear to be the same throughout the samples. In addition to the observed characteristics, each sample appears to have the same degree of chalking. Several samples were selected for further examination.

A second phase of the preliminary investigation was to separate samples into components to determine the approximate percentages of acid soluble and insoluble (fines) components in the binder. The acid soluble and insoluble (fines) components are portions of the cement binder and the fines are typically clays or cement residue. This investigation is primarily focused on the cement binder material to determine whether there are clays present. More specifically, the presence of significant amounts of clays within the binder could indicate that the John E. Park formula was used; if it is not present, it could indicate that the material is "limecrete," suggesting that the binder is from relatively pure lime. Lastly, if clay is present in the binder, but it is not of the percentage that John E. Park specifies, it could indicate that limestone with high clay content was used as a raw material.

After the representative samples were acid digested, each of the samples had about 10-13% of their initial weight calculated to be fines, or assumed clays. The acid soluble component was 75-85%. The fines were color matched and all had similar color, only varying slightly in one sample. Based on this preliminary investigation, the samples appear to contain a clay component in the binder. The similarity in sample color and binder color provides further indication that the concrete samples were constructed from materials made from the same or similar sources. At this point in the investigation, these assumptions were hypothetical; however, scanning electron microscopy with electron dispersive x-ray spectroscopy analysis will help to determine the chemical composition of the binder.

SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) is a technique that is commonly used for analysis in geology, biology, and the material sciences. Many scientists prefer to use SEM for imaging because it is less expensive than other instrumental techniques. SEM can be used with an energy dispersive x-ray spectroscopy (EDS) detector to identify elements and produces results within seconds.

For this investigation, representative samples were selected from the Moses Campbell commercial building, the Old Baptist Church, the Riverside Cemetery wall, and Sebastopol. Each of the samples was prepared by pulverizing and sieving the concrete to disaggregate the binder. Once the binder was isolated it was then applied to labeled specimen stub with carbon tape. SEM EDS detection provided information about the chemical composition of the samples based on the characteristic x-rays that are detected. For this analysis, a JEOL JSM-6490 LV with EDS detector was used. The operating conditions for EDS analysis were low vacuum mode, 20 Pascal pressure, 15 kV accelerating voltage, 10 mm working distance, spot size was 50, the tungsten filament current was 72 μA, and a count time of 100 seconds. For imaging, the experimental conditions were high vacuum mode, 20 kV accelerating voltage, 10 mm working distance, spot size was 50, and the tungsten filament current was 76 μA. BSE detector was selected with the shadow function.

Because gold coatings interfere with the analysis by blocking signals, the samples were left uncoated (non-conductive) for SEM EDS analysis. In order to carry out the SEM EDS analysis, the instrument must first capture a reference image in order for the user to determine which spots or areas to analyze with the data processing software. The SEM captures a BSE (Backscattered-electron) image and then imports it into the software. The images are captured in low-vacuum mode and these lower resolution images are automatically added to the EDS spectra.

After capturing the SEM EDS spectra, the samples were gold coated using a Denton Vacuum Desk II Sputter coater to make them conductive. The gold coated specimens were analyzed by SEM under high vacuum mode in order to capture high resolution images.   For the purpose of this investigation a solid-state backscattered-electron detector (SSD BSE) was selected. The BSE detector provides topographic or compositional information through the contrast of the image. When both sections of the SSD BSE detector are selected, also known as a combination contrast mode, an image is produced that provides both compositional and topographical information. This investigation used the BSE shadow image to provide a graphic representation of the information received during the SEM EDS analysis. The Au-coated, conductive samples were captured in high vacuum mode and provided higher resolution images.

EDS spectra were collected for each sample by selecting spots to analyze. At least ten spots were analyzed per sample, and the analyses varied only slightly within each sample. Each spectrum had similarly proportioned chemical elements of carbon, calcium, and oxygen; each sample also had smaller amounts of aluminum and silicon. Based on these spectra, the chemical elements detected indicate that the composition of the binder is most likely calcium carbonate ($CaCO_3$). The smaller concentrations of aluminum and silicon indicate that clay might be part of the binder. Although the presence of clays was not conclusively determined using SEM EDS, the spectra provided a good indication that clays were present in trace amounts. Additional analysis of the samples with X-ray diffraction helps to identify phases present on the basis of their crystalline structure, and determine if clay is present in the cement binder. The method should detect clays at concentrations above a few tenths weight percent, depending on the clay type.

X-Ray Diffraction

X-ray diffraction (XRD) is a technique used by mineralogists and materials scientists to examine the crystal structure of an unknown specimen. Analysis of the crystal structure aids identification of the chemical compounds present in a sample. Collected data are compared to reference standards containing the chemical elements identified through SEM EDS. For this reason, it is common for a researcher to use both XRD and SEM EDS for materials analysis.

For this investigation, the Bruker D8 Advance had a copper anode (λ_{α} = 0.15418 nm) and was equipped with a standard scintillation detector. The sample stage is fixed, but the x-ray tube and scintillation detector are located on arms that rotate around the sample stage. The experimental conditions used for this analysis were a Start position of 10° 2θ, Stop position of 80° 2θ, step size/increment 0.02°, time per step 0.5 seconds, generator voltage 40 kV, and generator current of 40 mA.

The Xray diffractogram patterns indicated that there is about 90-93% calcite (or calcium carbonate) and about 5-7% quartz present in each sample. Additionally, some of the samples contained a trace of gypsum. These results are consistent with the EDS results in which the samples contain similarly proportioned chemical elements of carbon, calcium, oxygen and also have smaller amounts of aluminum and silicon. The percentages were acquired with MDI Jade v.8 software in consultation with Dr. James Murowchick (University of Missouri-Kansas City) who used reference intensity ratio (RIR) values to approximate percentages for the phases present. RIR values are helpful in providing information about major phases in a sample, but can vary 5-10% over or under the approximated value. For our purposes, this could drastically skew the ratio between calcium carbonate and quartz. We suspected that clay or aluminum silicates would be present in the diffractogram data, but they were not observed. One possible explanation for absence is that the aluminum silicates were below the detectable limit of the instrument. Of course, the burning of clays at certain temperatures would destroy the crystalline structure and result in an amorphous mixture, or metakaolintite.[41] Based on the potential for RIR percentages to be inaccurate representations, we relied on the data from SEM EDS and the initial gravimetric analysis.

[41] A. Elena Charola and Fernando M.A. Henriques, "Hydraulicity in Lime Mortars Revisited," in *PRO 12: International RILEM Workshop on Historic Mortars: Characteristics and Tests*, ed. Peter Bartos, Caspar Groot, and John J. Hughes (RILEM Publications SARL, 1999), 97-99.

Chapter V: Conclusion

We set out to determine the composition of the cement binder used during the concrete construction era in Seguin. We have used historic, archival, and scientific research to investigate the materials used in the concrete construction. Three theories were identified in research:

1. Dr. Park's patented formula for a hydraulic cement binder was used
2. Dr. Park invented Limecrete, a misnomer
3. Raw materials were quarried locally and used

The first theory claims that Dr. John E. Park was the proponent and inventor of the formula for the Seguin concrete. Dr. Park's patented formula was for a hydraulic cement binder that called for 20-30% clay added to limestone in the calcination process. The second theory conflicts with the first; it attributes Park as the inventor of the concrete, but they call it Limecrete. Limecrete suggests that a relatively pure limestone was used as a raw material for the binder; this conflicts with Park's patent for hydraulic cement binder. The last theory is that raw materials for the binder were quarried locally and calcined without additional materials. At the outset, these conflicting theories provided us with a set of unknowns. We did not know the composition and the component percentages of the concrete's binder or that of the locally available raw materials.

After collecting representative samples from extant structures, we examined representative samples were similar in binder color and other physical characteristics. We used acid digestion as a means to separate the binder into acid soluble and non-soluble components. During this part of the preliminary investigation, it was determined that the representative samples all had 75-85 wt % acid soluble and 10-13 wt % acid insoluble components of the binder. SEM EDS analysis followed the preliminary investigation and indicated that the composition of the samples is most likely calcium carbonate ($CaCO_3$) with averages of 3wt % aluminum and 6 wt % silicon. These small amounts could indicate the presence of clays within the binder, represent impurities of the raw materials, or be signals picked up from the specimen stub during analysis. X-ray diffraction was conducted in order to identify the chemical compounds and crystalline structure of the binder. The results indicated that the representative samples had 92-93% calcium carbonate (calcite) and 5-7% quartz. If clays were present, we would have expected to see kaolinite (aluminum silicates) in the diffractograms. If kaolinite was fired during calcination, its crystal structure would have collapsed and would then be an amorphous metakaolinite. Amorphous materials are not detectable using XRD. The kaolinite pattern was not present in our diffractograms, meaning that it could either not be present or be under the detectable limit. Even if under the detectable limit, these percentages were still lower than those expected for Park's patented mix.

During the site visit in May, two samples were collected from the pit on the Hoermann property. Assisted by Federico Aguayo with the Construction Materials Research Group at the University of Texas at Austin, the samples were analyzed using Bruker D8 Advance XRD. We found that the two samples of limestone had diffractograms similar to the binder samples.[42] When comparing the results of the materials analysis, the composition of the local geological deposits, and the analyses of the samples collected from the pit found in proximity to concrete structures it is plausible that the concrete in Seguin is composed of lime binder made from locally quarried limestone. Although Dr. Park was in Seguin during the time of the concrete era, the

[42] Appendix F includes the laboratory report for X-ray Diffraction

cement binder does not appear to be Park's patented hydraulic cement. Additionally, his direct participation in construction is still not well documented; most of his activities have been suggested through oral histories.

The remaining concrete structures are at-risk due to deferred maintenance and improper repairs. Immediate next steps would be to recommend that extant structures be researched, examined, described and documented; this documentation could be a foundation for a multiple property nomination for the National Register of Historic Places. Also, a recommendation to the city and the Main Street Coordinator that repair and replacement material is in kind; more specifically that Portland cement stucco not be used on the structures. Each owner should also have a condition assessment completed for their building to understand its vulnerabilities. The assessment will also identify methods to be used to preserve the structure.

Appendix A

DR. JOHN E. PARK PATENTS

127,264

UNITED STATES PATENT OFFICE.

JOHN E. PARK, OF RUTHERFORD COUNTY, TENNESSEE.

IMPROVEMENT IN THE MANUFACTURE OF ARTIFICIAL STONE.

Specification forming part of Letters Patent No. 127,264, dated May 28, 1872.

SPECIFICATION.

Be it known that I, JOHN E. PARK, of Rutherford county and State of Tennessee, have invented a certain Process for Manufacturing Artificial Stone, of which the following is a specification:

Take, of the calcareous beds of Western Texas, and, where the percentage of clay is not equal to twenty-five, add to it until you make the percentage twenty-five or thirty. The mass is now moistened with salt-water at the rate of two per cent., using water enough to bring it to a proper consistency in a pug-mill. This material is molded into any shape or size, placed upon the yard to dry, same as brick, and when thoroughly dried, hacked up the same as brick, having a series of flues from bottom to top at intervals of two and a half feet. The kiln is now ready to be fired. Commence with the bottom tiers or series of flues; fire up slowly until the heat has obtained a cherry-red; hold it to this degree of heat four hours, being very careful not to let it reach a higher degree of heat, as if so, the mass becomes calcined and will slack down. The degree of heat aimed at is the development of incipient cementation. It may be known when incipient cementation is developed by the taste, not properly caustic, but slightly sweetish alkaline.

Claim.

1. I claim the compounding and burning of cementitious elements in such a manner as to produce incipient cementation in the block molded, after the manner specified.

2. Also, the form and construction of kiln.

J. E. PARK.

Witnesses:
C. W. MCGUIRE,
THOS. C. MCGUIRE.

Transcription:
United States Patent Office
John E. Park, of Rutherford County, Tennessee.
Improvement In The Manufacture of Artificial Stone.
Specification forming part of Letters Patent No. 127,264, dated May 28, 1872.

Be it known that I, John E. Park, of Rutherford county and State of Tennessee, have invented a certain Process for Manufacturing Artificial Stone, of which the following is a specification:

Take, of calcerous beds of Western Texas, and, where the percentage of clay is not equal to twenty-five, add it until you make the percentage twenty-five or thirty. The mass is now moistened with salt-water at the rate of two per cent, using water enough to bring it to a proper consistency in a pug-mill. This material is molded into any shape or size, placed upon the yard to dry, same as brick, and when thoroughly dried, hacked up the same as brick, having a series of flues from bottom to top at intervals of two and a half feet. The kiln is now ready to be fired. Commence with the bottom tiers or series of flues; fire up slowly until the heat has obtained a cherry-red; hold it to this degree of heat four hours, being very careful not to let it reach a higher degree of heat, as if so, the mass becomes calcined and will slack down. The degree of heat aimed at is the development of incipient cementation. It may be known when incipient cementation is developed by the taste, properly caustic, but slightly sweetish alkaline.

UNITED STATES PATENT OFFICE.

JOHN E. PARK, OF RUTHERFORD COUNTY, TENNESSEE.

IMPROVEMENT IN CEMENT-KILNS.

Specification forming part of Letters Patent No. 133,664, dated December 3, 1872.

To all whom it may concern:

Be it known that I, Dr. JOHN E. PARK, of the county of Rutherford and State of Tennessee, have invented a Cement-Kiln and Process of Burning, of which the following is a specification:

Cement-kilns are ordinarily defective in respect of the arrangement of base-flues or furnaces, whereby the material to be burned is unequally heated or acted on by the flame.

My improvement consists in arranging the flues so as to traverse the whole width of the kiln and open alternately at one side and the other.

In the accompanying drawing, Figure 1 is a perspective view of a kiln; and Fig. 2, a plan view, showing my improved arrangement of furnace-flues.

The base-flues A extend transversely across the kiln, and open or have their mouths alternately on opposite sides thereof. Thus the cement material will be equally heated whether it lie near one side of the kiln or the other, and the result of such action of the flame is a product of a homogeneous nature in place of the imperfect one obtained under the old arrangement of flues, in which the material on one side of the kiln was subjected to a heat much more intense than that on the other, whereby the cementitious quality of the same is often greatly impaired. In relation to hydraulic cement and its uses this result is highly important.

I have shown a series of upper flues, B, and watch-holes near the same, built with hearths projecting into the interior of the kiln.

The advantage of such provision of supplementary furnaces is obvious, as regards the proper heating of the mass of material removed from the immediate or most powerful influence of the heat or flame of the base-flues A.

The form of kiln is preferably rectangular and oblong.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

The arrangement of the base-flues or furnaces A, the same traversing the kiln and opening alternately on opposite sides thereof, as and for the purpose specified.

In testimony that I claim the foregoing as my own I affix my signature in the presence of two witnesses.

JOHN E. PARK.

Witnesses:
ROBERT McCOY,
E. F. WILSON.

Transcription:
United States Patent Office
John E. Park, of Rutherford County, Tennessee
Improvement In The Manufacture of Artificial Stone
Specification forming part of Letters Patent No. 133,664, dated December 3, 1872.
To all whom it may concern:

Be it known that I, Dr. John E. Park, of the county of Rutherford and State of Tennessee have invented a Cement Kiln and Process of Burning, of which the following is a specification:

Cement-kilns are ordinarily defective in respect to the arrangement of base-flues or furnaces, whereby the material to be burned is unequally heated or acted on by the flame. My improvement consists in arranging the flues so as to traverse the whole width of the kiln and open alternatively at one side and the other. In the accompanying drawing, Figure 1, is a perspective view of a kiln; and Fig. 2, a plan view, showing my improved arrangement of furnace-flues. The base-flues A extend transversely across the kiln, and open or have their mouths alternately on opposite sides thereof. Thus the cement material will be equally heated whether it lie near one side of the kiln or the other, and the result of such action of the flame is a product homogeneous nature in place of the imperfect one obtained under the old arrangement of flues, in which the material on one side of the kiln was subjected to a heat much more intense than that on the other, whereby the cementitious quality of the same is often greatly impaired. In relation to hydraulic cement and its uses this result is highly important. I have shown a series of upper flues, B, and watch-holes near the same, built with hearths projecting into the interior of the kiln. The advantage of such provision of supplementary furnaces is obvious, as regards the proper heating of the mass of material removed from the immediate or most powerful influence of the heat or flame of the base flues A. The form of kiln is preferably rectangular and oblong. Having described my invention, what I claim as new, and desire to secure by Letters Patent, is–The arrangement of base-flues or furnaces A, the same traversing the kiln and opening alternately on opposite sides thereof, as and for the purpose specified. In testimony that I claim the foregoing as my own I affix my signature in the presence of two witnesses.

J. E. PARK.

Cement-Kiln.

No. 133,664. Patented Dec. 3, 1872.

Fig.1.

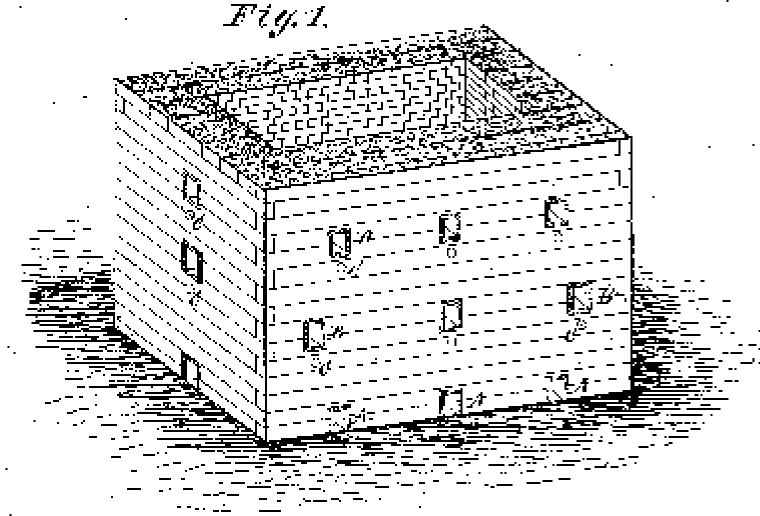
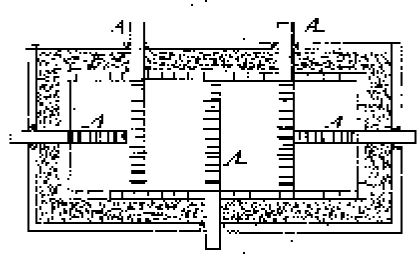


Fig.2.



Witnesses:

Inventor:
John E. Park

Per Munn & Co

Attorneys.

UNITED STATES PATENT OFFICE.

JOHN E. PARK, OF LA VERGNE, TENNESSEE.

IMPROVEMENT IN THE MANUFACTURE OF CEMENT.

Specification forming part of Letters Patent No. **138,924**, dated May 13, 1873; application filed April 6, 1871.

To all whom it may concern:

Be it known that I, JOHN E. PARK, of La Vergne, Rutherford county, Tennessee, have invented a new and Improved Cement, and process of burning it, of which the following is a specification:

My invention relates to a new hydraulic cement, and to a process of burning the same to develop its highest cementitious qualities. The cement is composed of any of the forms of lime, with thirty to forty per cent. of clay, (alumina and silica,) five to ten of fine sand, (silex,) and five per cent. of soda (carbonate, muriate, or caustic) or potash. These materials are mixed together and moderately worked into union so that they will not be in very intimate contact; the less the amount of clay the more intimate should be the mixture, and the larger the amount of clay the less intimate. The mass thus formed is first dried, then broken up and burned in a kiln provided with inspection or watch holes, and furnaces or fire-places, one above another, or at various heights from its base, to enable the burner to know the exact condition of, and have perfect control over, the burning at all times. The material is stratified with wood, or other combustible material, and the mass brought to a full red heat. If now a specimen be withdrawn from one of the watch-holes and broken open, it will show a pale-blue color, which indicates insufficient calcination. The heat must be continued until the specimens thus withdrawn present a bright sulphur color, both externally and internally, and the surface of the mass in the kiln shows incipient fusion or vitrification. These are the marks of a perfect calcination such as will alone produce a cement of the best or most highly cementitious character, but incipient surface fusion is the main reliance, since the materials of the cement will sometimes have different proportions of metallic oxides in their composition and the color vary a little under the influence of heat. No good cement can be produced by any degree of heat as indicated by a thermometer or pyrometer, as different compounds are more or less vitreous; the above marks are the guides. As long-continued heat at a lower temperature is prejudicial to the quality of the cement the burn should be finished in from eight to fifteen hours.

The proportions of the ingredients of the cement may vary somewhat, but I prefer those above given as being the result of numerous experiments and practical tests. The lime or cement thus produced will slack with water and will not set in water, but will set immediately in the air; the hardening process will then go on in water.

It may be remarked that the substantially same tests of calcination apply to the burning of a water-setting cement composed of lime with clay, fifteen to twenty-five per cent.; fine sand, three to five per cent.; and soda or potash, three to five per cent.

Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The hydraulic lime or cement composed of the ingredients in the proportions specified.
2. The process of preparing and calcining the cement, as set forth.

JOHN E. PARK.

Witnesses:
ROBERT McCAY,
E. F. WILSON.

United States Patent Office
John E. Park, of La Vergne, Tennessee.
Improvement in the Manufacture of Cement
Specification forming part of Letters Patent No. 138,924, dated May 13, 1873; application filed April 6, 1871.
To all whom it may concern:

Be it known that I, John E. Park, of La Vergne, Rutherford County, Tennessee, have invented a new and Improved Cement, and process of burning it, of which the following is a specification:

My invention relates to a new hydraulic cement, and to a process of burning the same to develop its highest cementitious qualities. The cement is composed of any of the forms of lime, with thirty to forty per cent. Of clay, (alumina and silica,) five to ten of fine sand, (silex,) and five per cent. Of soda (carbonate, muriate, or caustic) or potash. These materials are mixed together and moderately worked into union so that they will not be in very intimate contact; the less amount of clay the more intimate should be the mixture, and the larger amount of clay the less intimate. The mass thus formed is first dried, then broken up and burned in a kiln provided with inspection or watch holes, and furnaces or fire-places, one above another, or at various heights from its base, to enable the burner to know the exact condition of, and have perfect control over, the burning at all times. The material is stratified with wood, or other combustible material, and the mass brought to a full red heat. If now a specimen be withdrawn from one of the watch-holes and broken open, it will show a pale-blue color, which indicates insufficient calcination. The heat must be continued until the specimen thus withdrawn present a bright sulphur color, both externally and internally, and the surface of the mass in the kiln shows incipient fusion or vitrification. These are the marks of a perfect calcination such as will alone produce a cement of the best or most highly cementitious character, but incipient surface fusion is the main reliance, since the materials of the cement will sometimes have different proportions of metallic oxides in their composition and the color vary a little under the influence of heat. No good cement can be produced by any degree of heat as indicated by a thermometer or pyrometer, as different compounds are more or less vitrescent; the above marks are the guides. As long-continued heat at a lower temperature is prejudicial to the quality of the cement the burn should be finished in from eight to fifteen hours.

The proportions of the ingredients of the cement may vary somewhat, but I prefer those above given as being the result of numerous experiments and practical tests. The lime or cement thus produced will slack with water and will not set in water, but will set immediately in the air; the hardening process will then go on in water.

It may be remarked that the substantially same tests of calcination apply to the burning of water-setting cement composed of lime with clay, fifteen to twenty-five per cent; fine sand, three to five per cent; and soda or potash, three to five per cent.

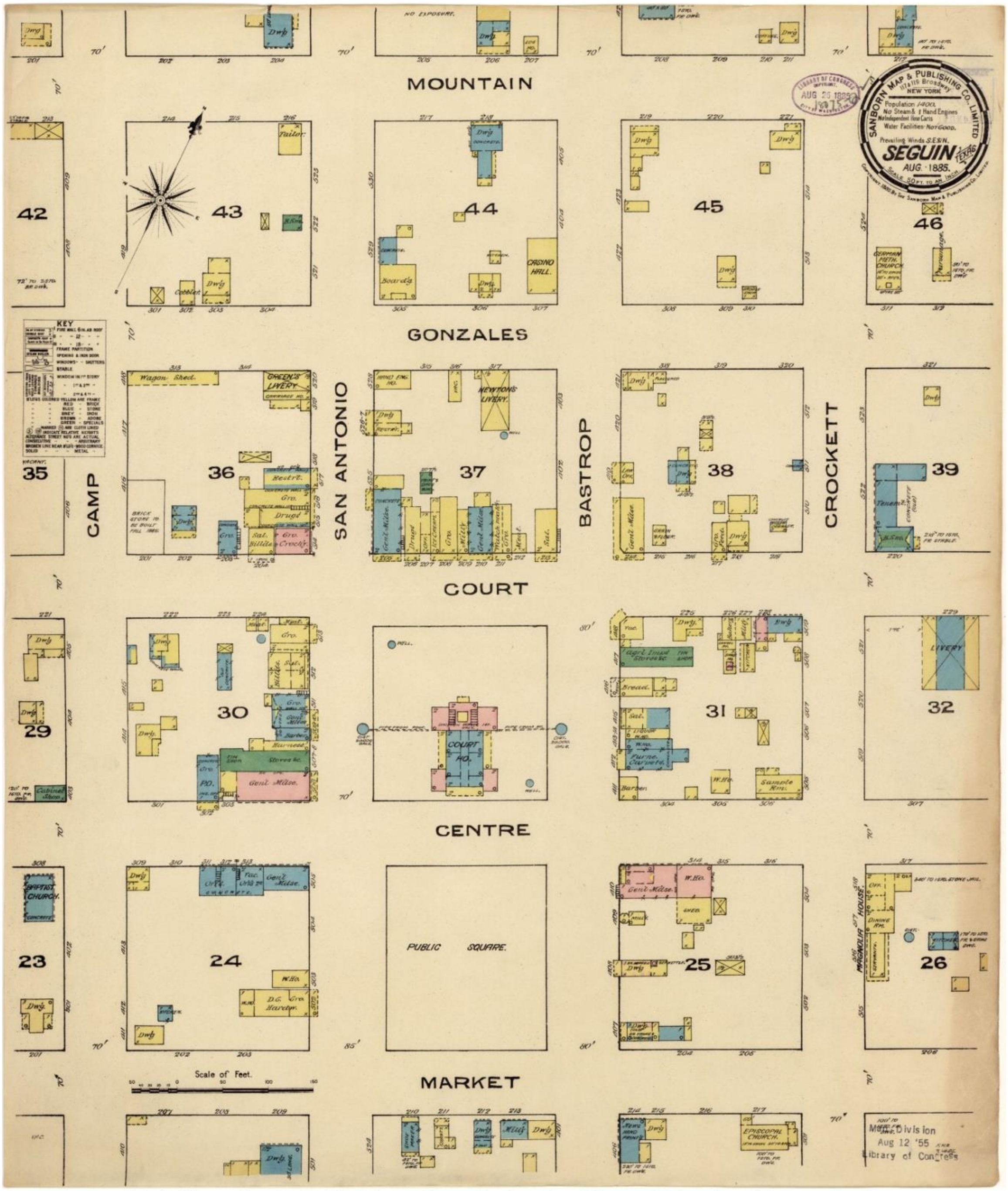
Having thus described my invention, what I claim as new, and desire to secure by Letters Patent, is–

1: The hydraulic lime or cement composed of the ingredients in the proportions specified.

2: The process of preparing and calcining the cement, as set forth.

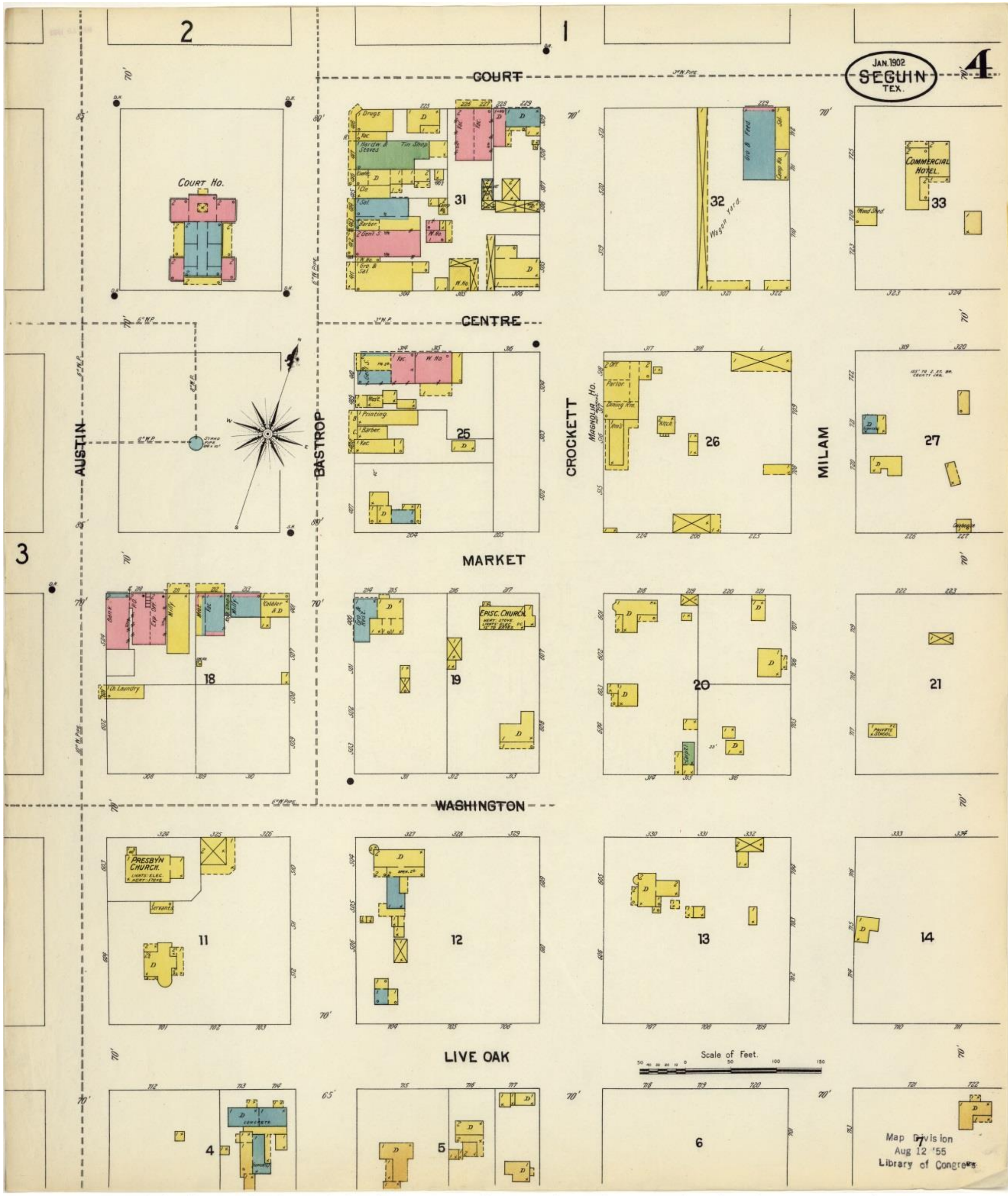
Appendix B

SANBORN MAP, SEGUIN, TEXAS, 1885 & 1902



Original located at the Dolph Briscoe Center for American History, University of Texas at Austin.

*The Riverside Cemetery wall and Sebastopol are not included on this map.



Original located at the Dolph Briscoe Center for American History, University of Texas at Austin.

*The Riverside Cemetery wall and Sebastopol are not included on this map.

Appendix C

LABORATORY REPORT: SAMPLE DESCRIPTIONS

ARCHITECTURAL CONSERVATION LAB
HISTORIC PRESERVATION PROGRAM
SCHOOL OF ARCHITECTURE
THE UNIVERSITY OF TEXAS AT AUSTIN

DATE: February 4, 2014
LABORATORY REPORT: Sample Descriptions
PREPARED BY: Sarah B. Hunter

PURPOSE: The purpose of this report is to compile initial descriptions of samples, identify the structures from which they are extracted, define attributes and describe potential causes of deterioration.[43]

SAMPLES: The samples were collected from several extant structures in Seguin, Texas on September 12, 2013 and September 18, 2013. An identification code was made based on the structure name, whether the samples were extracted from the interior or exterior of the structure, and a numerical value to differentiate between samples that shared the same first two characteristics.

The sample selection criteria were to gain permission from building owners to extract samples from structures that had accessible original material (exposed, not encapsulated), that the structure be constructed c. 1850-1880, and that the structure was in reasonable condition. In all structures, it was challenging to keep samples intact upon extraction and transportation due to the conditions in the material. The table below shows the structures that were included:

[43] This report begins with a recap of Chapter IV.

Structure & construction date	Photograph	Sample numbers	Notes
Moses Campbell Commercial building 104 N. Austin c.1850		AI1, AI2	Structural wall within the commercial building, lower half is caliche blocks, upper portion is concrete; interior, chalking present
Old Baptist Church 1880		BE1, BE2, BE3, BI1	Concrete material is from the exterior and interior, differing weathering conditions, but chalking is present throughout
Riverview Cemetery Wall c.1850		CE1, CE2	Material is exposed to exterior; chalking; no evidence remaining of stucco
Magnolia Hotel c. 1847		MI1	Material is exposed around single window frame in interior walkway between concrete and adjoining wood frame structure; limited area of access

Sebastopol 1854		SI1, SI2, SI3	Material is only exposed in interior spaces in basement; renovated in 1980; chalking

DETERIORATION: The following are terms used to describe concrete conditions. As aforementioned, the material collection in each structure was challenging as it was friable. In the terms described below, moisture was likely a factor. Moisture can be from wind-driven rain, water-vapor, or rising damp. Ultimately, moisture infiltrates the building envelope, eventually causing deterioration. Resulting conditions are described below.

Chalking: Formation of a loose powder resulting from the disintegration of the surface of concrete

Friable: Easily crumbled and becomes powder in form

Delamination: A term to describe a condition in stucco. A separation along a plane parallel to a surface, a horizontal splitting, or separation within the slab in a plane roughly parallel to, and generally near, the upper surface

Gypsum[44]: Reactions with sulfates that transform calcium carbonate into calcium sulfate dihydrate, leading to physical and mechanical changes in concrete

SAMPLES: The following are the samples used in this study. They are grouped under the structure that they were extracted from. In all cases, the material was friable and crumbled upon extraction. If samples contained large aggregate, it was gray in color:

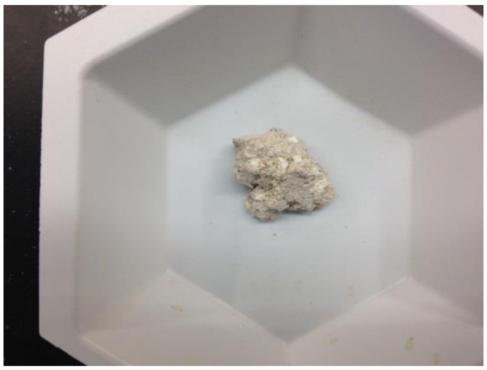
<u>Moses Campbell Commercial Building</u>

The samples were extracted from a concrete wall that was enclosed within the building on the first floor. During remodeling efforts, the wall was exposed and partially demolished. The portions that remain are supported by steel beams and columns and exposed to the interior space. The concrete wall is 19” in width and has pour lines at 12” intervals. This wall has large caliche[45] towards the bottom, but was constructed of concrete at 3’ and above. All of the samples were chalking.

[44] This is not a deterioration condition, but a deterioration product.

[45] Caliche stones are flat rectilinear limestone blocks.

AI1: The sample was extracted on the eastern section of the wall. It is light brown-gray in color with trace amounts of white chunks that appear to be lime. The material has large aggregate that is gray in color.



AI1

AI2: The sample was extracted on the western section of the wall. It is light brown-gray in color with trace amounts of white chunks that appear to be lime. The material has large aggregate that is gray in color.

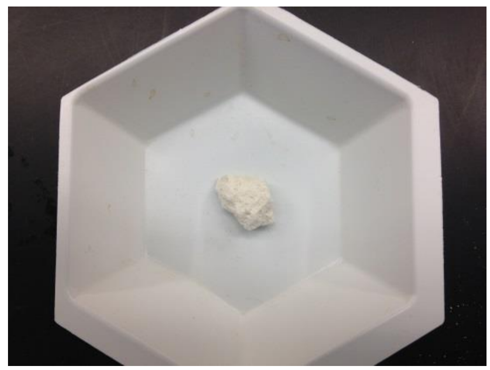
AI2

Old Baptist Church

The samples were extracted from various locations within the interior and exterior of the structure. When sampling from exterior exposed areas, we tried to identify areas that were intact. This structure has a 20th century stucco finish, which was likely a replacement.

BE1 & BE2: These samples were collected from the exterior of the Old Baptist Church. The second floor of the south elevation could be accessed from the roof of an addition. The area had substantial loss of stucco and the original concrete material was exposed. Exact wall thickness could not be determined from the exterior, but distinct pour lines were 12” apart. The samples were extracted from the exposed area on the wall, BE1 was from the upper portion and BE2 was from the lower. It is light brown-gray in color. The material has large aggregate intact in the sample.

BE1



BE2

BE3: This sample was collected from the exterior of the Old Baptist Church. There is a two story tower located on the Northeast of the structure, prior to our visit, a large portion of the tower’s stucco detached and was saved for our investigation. The stucco is made of 20th century cement and wrapped the corner of the tower. When it detached, it had a large portion of original material adhered to the inner surface. This material is identified as original by its characteristics of chalky binder and coarse aggregate and the evidence of distinct pour lines at 12” intervals. Exact wall thickness could not be determined from this section. The sample was extracted from the exposed area. It is light brown-gray in color. The material has large aggregate intact within the sample.

BE3

BI1: This sample was collected from the interior of the Old Baptist Church. Inside of the northeast tower is a small entryway with an accessible junction box. When the cover is removed, the wall thickness can be measured. The wall has an approximate width of 15”. The sample was extracted from the exposed area. It is light gray/ chalky white in color. The material has large aggregate that is intact in the sample.

BI1

Riverside Cemetery Wall

The following samples were collected from the Riverside Cemetery walls. Isolated in sections, the wall once surrounded the cemetery. The wall has small areas of what appears to be stucco, but it is now deteriorated.

CE1: In the southernmost part of the cemetery, two sections stand while others have fallen over but otherwise remain intact. The walls are 15” in thickness, but do not have visible pour lines. The sample was extracted from the lower portion of a connection joint on the east elevation. It is light gray in color with a yellow tinge. The material has large aggregate intact in the sample.

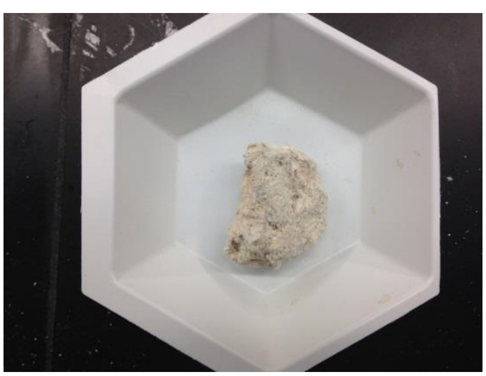
CE1

CE2: In the southernmost part of the cemetery, two sections stand while others have fallen over but otherwise remain intact. The walls are 15” in thickness, but does not have visible pour lines. The sample was extracted from the upper portion of a connection joint on the east elevation. It is light gray in color with a yellow tone. The material has large aggregate intact in the sample.

CE2

Magnolia Hotel

MI1: This sample was collected from the interior of the Magnolia Hotel. The hotel was constructed in two phases, the first being a one story concrete structure with a later two-story wood framed addition to the north. The later addition served as the primary hotel, while the concrete portion of the hotel became servant’s quarters. The one story structure was recently renovated, only a portion of the wall material was exposed next to a window in the corridor that attached the concrete structure to the wood frame structure. The walls are 8” in thickness, but pour lines could not be determined. The sample was extracted from the exposed area on the west facing interior elevation. The material was friable and crumbled upon extraction. It is light gray in color with a yellow tone. The material has large aggregate that is intact in the sample.



MI1

Sebastopol

The following samples were collected from the interior of Sebastopol. Sebastopol is a raised one story structure with access to ground level basement. The structure has undergone several renovations and is the best documented of all the structures in this report. Original material is left exposed in the interior basement space. In this basement area walls are 15" in width and have pour lines at 12" intervals. Some of the walls towards the exterior of the building have been reconstructed and repaired.

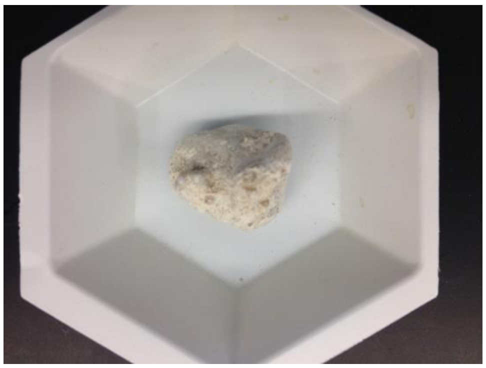
SI1: This sample was collected from the interior of Sebastopol. The sample was extracted from the east wall in the western interpretation room. Has what appears to be either gypsum or chalking. It is light brown-gray in color. Its large aggregate is no longer intact within the sample.

SI1

SI2: The sample was extracted from the east wall in the center room. Has what appears to be either gypsum or chalking. It is light brown-gray in color with large aggregate that is intact within the sample.

SI2

SI3: The sample was extracted from the south wall in the secret room. Has what appears to be either gypsum or chalking. It is light brown-gray in color with large aggregate intact within the sample.



SI3

Appendix D

LABORATORY REPORT: PRELIMINARY EXAMINATION

ARCHITECTURAL CONSERVATION LAB
HISTORIC PRESERVATION PROGRAM
SCHOOL OF ARCHITECTURE
THE UNIVERSITY OF TEXAS AT AUSTIN

DATE: February 4, 2014
LABORATORY REPORT: Preliminary Exam
PREPARED BY: Sarah B. Hunter

PURPOSE OF TESTING:

This preliminary testing includes an initial visual observation and color matching of the binder. The observation of physical characteristics informs our decisions when selecting representative samples. After representative samples are selected, the samples are separated into components. The acid soluble component is digested, leaving the aggregate and acid insoluble material (fines or clays) of the sample. Aggregate shape, size, and color are noted and the approximate weight percentage of each component is calculated. The testing will assist in determining composition of the sample and enable us to make assessments about the materials.

SAMPLES:

The samples were collected from several extant structures in Seguin, Texas on September 12, 2013 and September 18, 2013. An identification code was made based on the structure name, whether the samples were extracted from the interior or exterior of the structure, and a number to differentiate between samples that shared the same first two characteristics.[46]

Sample ID: AI1
Weight: 30 g
Structure: Moses Campbell Commercial Building
Location: Interior

Sample ID: AI2
Weight: 45 g
Structure: Moses Campbell Commercial Building
Location: Interior

Sample ID: BE1
Weight: 42 g
Structure: Old Baptist Church
Location: Exterior

Sample ID: BE2
Weight: 43 g
Structure: Old Baptist Church
Location: Exterior

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[46] For more information, see the appendix "Sample Descriptions".

Sample ID: BE3
Weight: >100 g
Structure: Old Baptist Church
Location: Exterior

Sample ID: BI1
Weight: 30 g
Structure: Old Baptist Church
Location: Interior

Sample ID: CE1
Weight: 33 g
Structure: Riverside Cemetery Wall
Location: Exterior

Sample ID: CE2
Weight: 42 g
Structure: Riverside Cemetery Wall
Location: Exterior

Sample ID: MI1
Weight: 22 g
Structure: Magnolia Hotel
Location: Interior

Sample ID: SI1
Weight: 30 g
Structure: Sebastopol
Location: Interior

Sample ID: SI2
Weight: 26 g
Structure: Sebastopol
Location: Interior

Sample ID: SI3
Weight: 35 g
Structure: Sebastopol
Location: Interior

TEST METHOD:

Concrete samples are examined in the Architectural Conservation Lab using a microscope, imaging system and color system.

MATERIALS AND EQUIPMENT:

10x loupe, variable magnification (10 - 63x) Nikon SMZ 800 Stereomicroscope equipped with fiber optic illumination and a Nikon NIS-Elements imaging system, Munsell Standard Color System, Ohaus electronic balance, 600 mL glass beaker, wash bottle, mortar and pestle, plastic graduated cylinder, dilute hydrochloric acid, filter paper circle, 500 mL Erlenmeyer flask, funnel

PROCEDURE:

Further examination of the mortar samples includes determining the weight percentage of the acid soluble, fines (cement and clay residue) and aggregate components. The acid digestion method is adapted from ASTM C1324 Standard Test Method for Examination and Analysis of Hardened Masonry Mortar. A portion of each sample is pulverized using a ceramic mortar and pestle, and then weighed. The sample is then moistened with water and dilute hydrochloric acid is slowly added to the pulverized samples to digest the acid soluble component. Following acid digestion, aggregate is separated from the fines using a filtration process. The aggregate and fines are allowed to dry and then each component is weighed and percent weights are calculated. The binder color of each mortar sample is matched to a color chip of the Munsell Color System, a standard system that specifies color in three dimensions: hue, value and chroma. Color matching is carried out in natural light.

Photomicrographs of each concrete sample and its fines component are included with the report of our examination.

<u>Examination</u>:

1. Select freshly broken sample of at least 20 g (if available)
2. Describe the sample
3. Use the Munsell color system to match the binder color

<u>Separation into components</u>:

1. Weigh the beaker using electronic balance and record weight
2. Pulverize sample using mortar and pestle. Note: do not use the entire sample
3. Transfer pulverized sample into glass beaker and take combined weight.
4. Subtract weight of beaker from the combined weight to determine the weight of the pulverized sample
5. Use wash bottle to moisten pulverized sample
6. Under fume hood, transfer small amount of 3 M hydrochloric acid to plastic beaker
7. Slowly add approximately 10 mL of 3 M hydrochloric acid to moistened sample
8. Record of observations, such as odor, reaction, and approximate amount of acid used
9. Continue adding small amounts of 3 M hydrochloric acid to dissolve the acid soluble material; between additions of dilute acid, carefully swirl the beaker to ensure complete reaction

10. When reaction is complete, slowly add 100 mL of water to dilute the solution in beaker
11. Weigh filter paper circle and record
12. Fold the filter paper into quarters by folding in half twice
13. Place folded filter paper into funnel to use as a liner, moisten liner with wash bottle to secure its placement
14. Place lined funnel on top of 500 mL Erhlenmeyer flask
15. Carefully swirl the diluted solution in the beaker to suspend the clay and cement residue not dissolved by the acid
16. Before settling, slowly pour the suspension into the funnel
17. Add more water to the suspension and repeat step 15-16 to complete collection of fines
18. Pour off as much liquid as possible while keeping aggregate in the beaker
19. Set beaker and flask aside to dry

Determining percent weight of components:

1. Weigh the filter paper with the dried “fines” and subtract preliminary weight of the filter paper to determine the “fines” weight
2. Examine the dried “fines” and color match using the Munsell Color System
3. Weigh the beaker and dried aggregate and subtract the beaker weight to determine the dried aggregate weight
4. Add “fines” and aggregate weight and then subtract the total from the original pulverized sample weight to calculate the weight of the acid soluble component
5. Calculate the percent weight for each component

TEST RESULTS

OBSERVATIONS:

The initial visual observations confirm that the samples are very similar in binder color, with only minute differences in detectable yellow value. Photomicrographs of each sample at 10 x magnifications enable us to see differences in overall color, aggregate color, shape and size, and soiling. It appears that samples extracted from unprotected exterior locations have more soiling and deposits. Overall, the distribution of fine aggregate is similar throughout the samples, binder color appears to be the same, and the exposed samples seem to have the same amount of chalking.

Color Matching Binder	
Sample:	Sample Color (Pre-Separation):
AI1	2.5Y 8/2
AI2	2.5Y 8/2
BE1	2.5Y 8.5/2
BE2	2.5Y 8.5/2
BE3	2.5Y 8.5/2
BI1	2.5Y 8.5/2
CE1	2.5Y 8/2
CE2	2.5Y 8/2
MI1	2.5Y 8/2
SI1	2.5Y 8.5/2
SI2	2.5Y 8/2
SI3	2.5Y 8/2

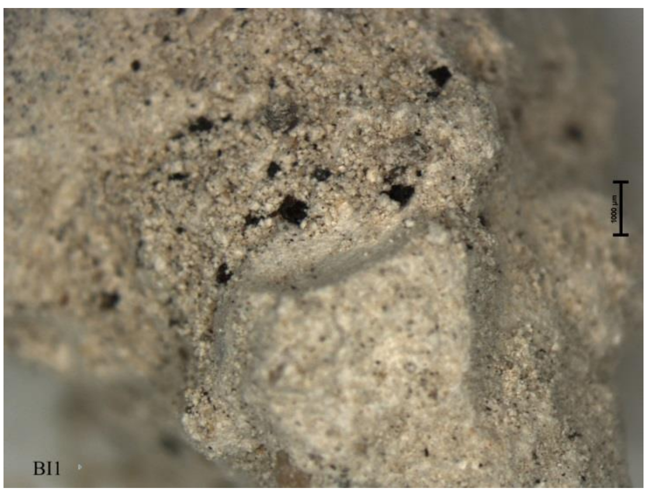
The following photomicrographs show each intact sample under magnification. You are able to see the physical properties of the binder and aggregate in each image. The black spots that appear in some of the samples are likely to be charred bits from the calcining process. The green-brown color present on some of the samples is biological growth.

AI1, 10x

AI2, 10x

BE1, 10x

BE2, 10x



BI1, 10x

CE1, 10x

CE2, 10x

MI1, 10x

SI1, 10x

SI2, 10x

SI3, 10x

ACID DIGESTION:

After the initial visual observation, it was apparent that the samples shared physical characteristics that made it unnecessary to separate every sample into components. Additionally, many of the samples turned to powder during extraction and transport leaving only small portions of the samples intact. Of the above listed samples, only four representative samples were selected to be separated into components. AI1, BE3, and CE1 were identified as representative samples and were selected for acid digestion. Below is a chart of the component percentages for the aforementioned samples.

Component Percentages:				
Sample	Initial Weight, g	Acid Soluble, g	Aggregate, g	Fines, g
AI1	7.8	6.6 (85%)	0.4 (5%)	0.8 (10%)
BE3	42.3	17.6 (42%)	20.5 (48%)	4.2 (10%)
CE1	16.7	12.7 (76%)	1.9 (11%)	2.1 (13%)

The component percentages of AI1 and CE1 are very similar due to similar amounts of large aggregate that remained in the sample prior to being pulverized. Sample BE3 was extracted from the large piece of stucco after transport and therefore was a larger intact sample size of 42.3 g. This sample included a higher amount of large aggregate, as it did not dislodge during transport. This could serve as an explanation of the differences in component percentages for acid soluble and aggregate. Additionally, the fines component in all three samples had similar percentages of acid soluble to acid insoluble material.

After the components are separated, we review the color of the fines in comparison with the other fines and the original binder color prior to acid digestion. AI1 and BE3 have the exact same color for their fines, while CE1 has only a slightly different color. The difference could be attributed to effects of hydrochloric acid on the fines.

Color Matching Fines in Comparison with Binder Color:		
Sample	Binder Color (Pre-Separation)	Fines Color
AI1	2.5Y 8/2	2.5Y 6/2
BE3	2.5Y 8.5/2	2.5Y 6/2
CE1	2.5Y 8/2	2.5Y 7/4

DISCUSSION OF RESULTS

Our investigation is primarily focused on the cement binder material to determine whether clay is present. If clay is found in the binder, it will help assess the plausibility of each of the theories surrounding the concrete construction in Seguin. More specifically, the presence of clay within the binder could indicate that John E. Park formula was used or if it is not present, it could indicate that the material is "limecrete", suggesting that the binder is relatively pure lime without additives.

Based on visual observation and laboratory testing, it is indicated that the samples may contain a clay component in the binder. The main indication of this is seen in the component percentages, where all three samples have 11-19% of the sample being 'fines' or our anticipated clay content. The similar color of the samples and fines suggest that the concrete samples could be constructed from materials taken from the same sources. While these assumptions remain hypothetical, additional testing would provide more specific answers about the samples. Using scanning electron microscopy with electron dispersive spectroscopy, we are able to determine the chemical composition of the binder, and expect to discover more information about the binder and its components.

Appendix E

LABORATORY REPORT: SCANNING ELECTRON MICROSCOPY

ARCHITECTURAL CONSERVATION LAB
HISTORIC PRESERVATION PROGRAM
SCHOOL OF ARCHITECTURE
THE UNIVERSITY OF TEXAS AT AUSTIN

DATE: February 4, 2014
LABORATORY REPORT: Investigation with Scanning Electron Microscopy
PREPARED BY: Sarah B. Hunter

PURPOSE OF TESTING: This investigation uses scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS) to determine the chemical composition of the cement binder taken from several extant structures in Seguin, Texas. SEM EDS analysis will identify the elemental components and provide weight percentages of those components in targeted areas, including trace elements that are present in the cement binder.

SAMPLES: Please refer to Appendix "Sample Descriptions" for more information regarding samples. Several representative samples were selected based on preliminary investigations and extracted from each structure to be included in this portion of the study. The samples included AI1, BE3, BI1, CE1, SI1, and SI3.

TEST METHOD: For this investigation, we used a scanning electron microscope (SEM) with energy dispersive X-ray spectroscopy (EDS) and back-scattered electron (BSE) imaging. All of the work was done in the University of Texas at Austin Jackson School of Geosciences E-beams Laboratory and was part of the Fall 2013 Course "Analytical Methods Electron Microbeam Technologies" (GEO390R). The SEM is a JEOL JSM-6490 LV. It is equipped with several detectors, but for the purposes of this investigation we used the EDS detector and the BSE detector. A BSE detector can collect an image in three modes; however, Shadow mode was selected for this analysis as it gives information about the composition and surface morphology through contrast and three dimensional topography. We used the Electron Microbeam Laboratories standard operating procedure for the SEM.

MATERIALS AND EQUIPMENT:
Sample Preparation: ceramic mortar and pestle, standard 3" diameter sieve set
Analysis: JEOL JSM-6490 LV with EDS detector, specimen stubs, carbon tape, specimen holder
Imaging: JEOL JSM-6490 LV with BSE detector, specimen stubs, carbon tape, specimen holder, Denton Vacuum Desk II Sputter (gold sputter coater)

PROCEDURE:
Sample Preparation: To analyze the cement composition, the binder had to be isolated from the aggregate prior to analysis. Each sample was pulverized using a ceramic mortar and pestle, separated using a 3" diameter sieve set, material collected in pan and sieve screen no. 100 & 200 were again pulverized, separated a second time in a standard sieve set, and material caught in the pan (less than 75 μm) was used for analysis. This method was the best way to isolate the binder with the equipment available. Once the samples were prepared, they were applied to specimen stubs using carbon tape and placed in the specimen holder (fig. 1).

EDS Analysis: For EDS, the samples were non-conductive and the following conditions were used: Low Vacuum mode, 20 Pascal pressure, 15 kV Accelerating voltage, 10 mm working distance, Spot size was 50 nm, the tungsten filament was 72 µA, and the z=7.999. The magnification was set to 2,000x in order to determine which spots to analyze. EDS analysis captures an image of the sample for use in the analysis software. This image allows the user to target either spots or entire areas for identification. Both functions were used to identify elements present in the sample. This BSE image will be lower resolution as the samples are non-conductive and we are operating in low vacuum mode.


Figure 1

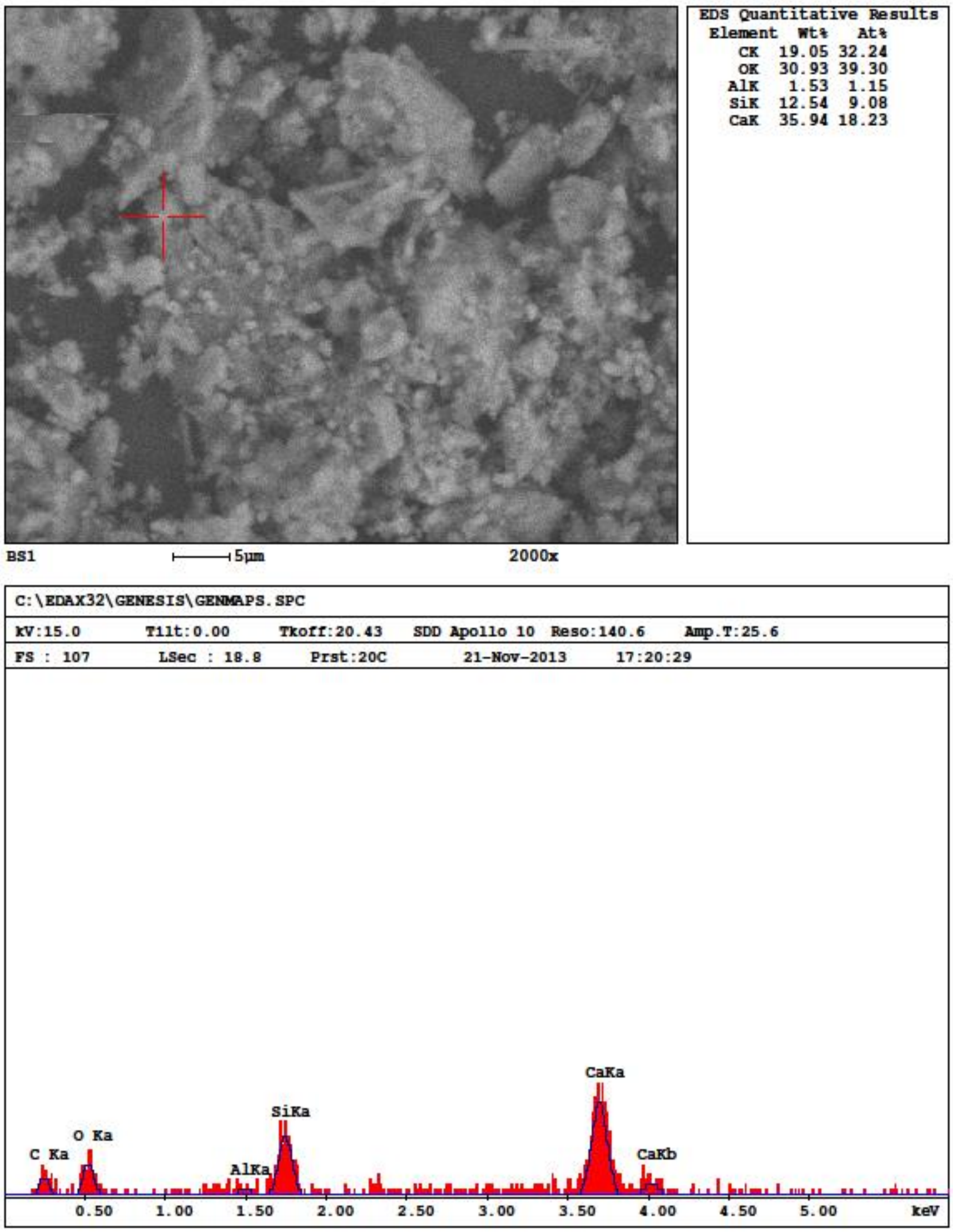
BSE Shadow Imaging: For SEM shadow BSE images, the samples were gold sputter coated and the following experimental conditions were used: High Vacuum mode, 20 kV Accelerating voltage, 10 mm working distance, Spot size was 50 nm, and the tungsten filament was 76 µA. BSE detector was selected with the shadow function.

TEST RESULTS:

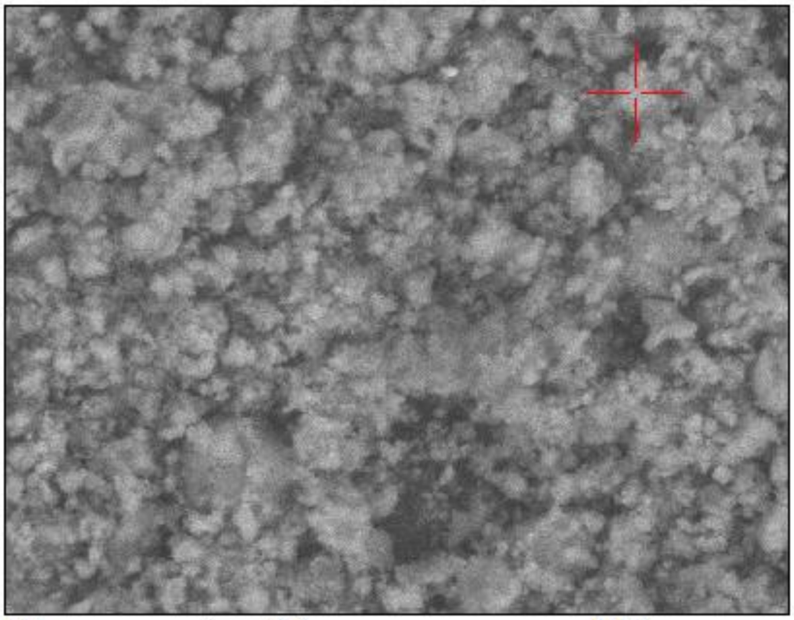
BSE Shadow Imaging:
The BSE images provide high resolution and high magnification details of the samples. The contrasts within the images are good indicators of the different phases (or elements) that are within the sample. The surface morphology can also be seen in the images and can be studied to understand particle size and the nature of the binder material.

EDS Analysis:
Based on EDS spectra, each sample had major elements of carbon, calcium, and oxygen; each sample also had aluminum and silicon present, but in smaller amounts. With over ten spots analyzed per sample, the spectra varied only slightly, but maintained similar proportions of the elements. The red crosshair on the image of each spectra indicate the target area for analysis. The images in the spectra are BSE Shadow images, with much lower resolution than the next set of images. This is due to the fact that the non-conductive samples were imaged in low vacuum mode. The following pages include the EDS spectra for each sample.

AI1-EDS Spectra:

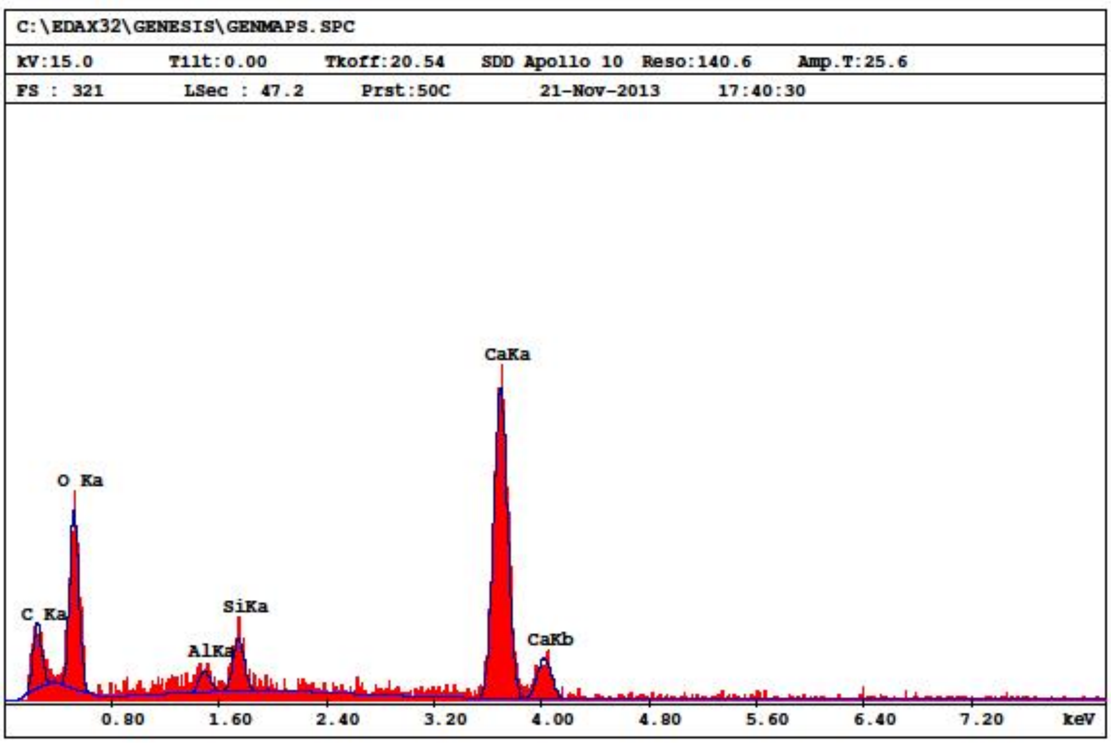


BE3-EDS Spectra:

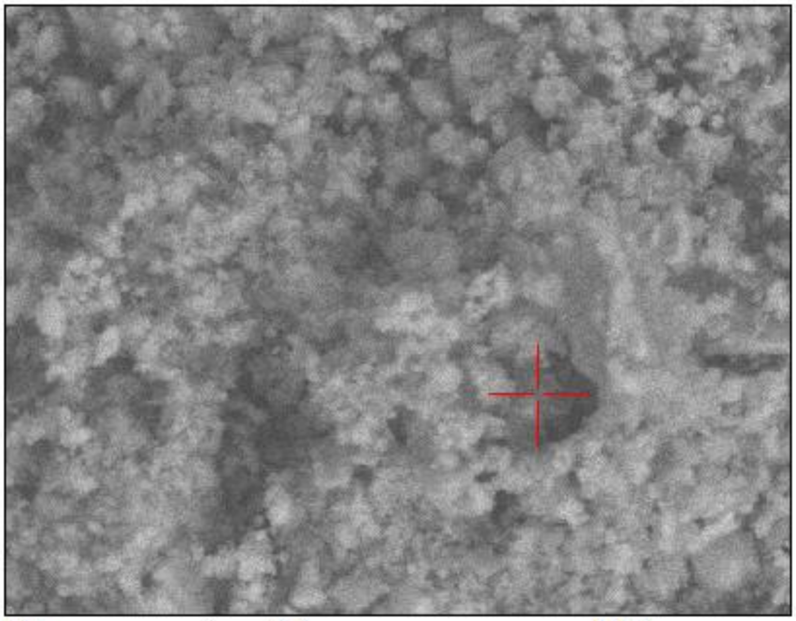


EDS Quantitative Results

Element	Wt%	At%
CK	15.43	24.86
OK	45.72	55.29
AlK	1.57	1.12
SiK	3.54	2.44
CaK	33.75	16.29



BI1-EDS Spectra:



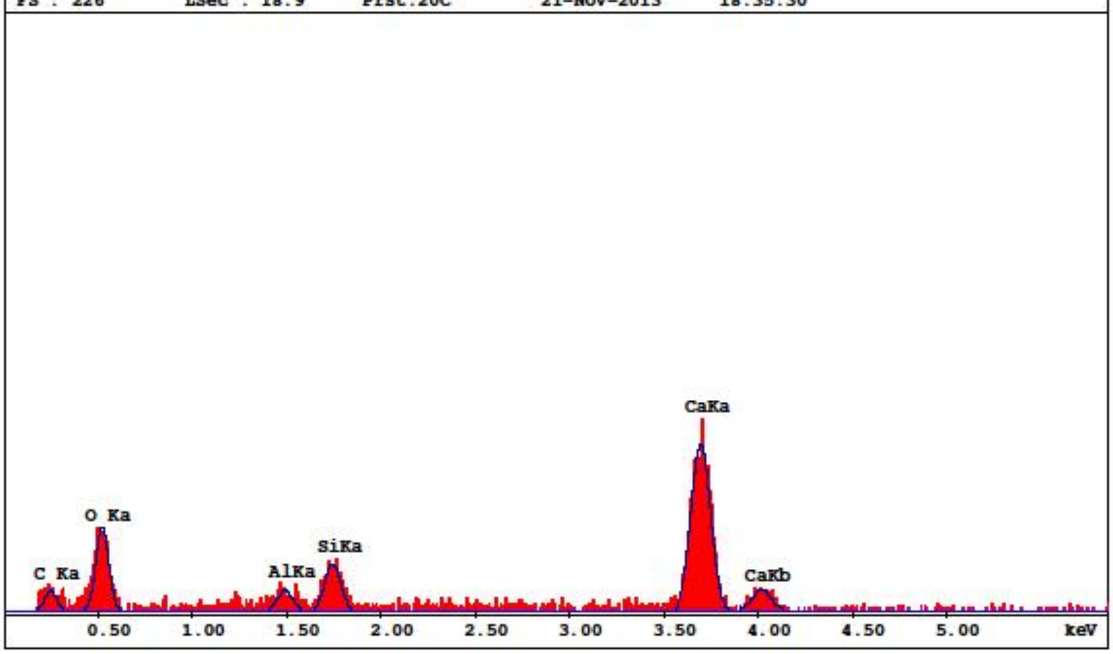
EDS Quantitative Results		
Element	Wt%	At%
CK	11.46	19.51
OK	43.31	55.35
AlK	2.80	2.12
SiK	6.31	4.59
CaK	36.12	18.43

BS1 5µm 2000x

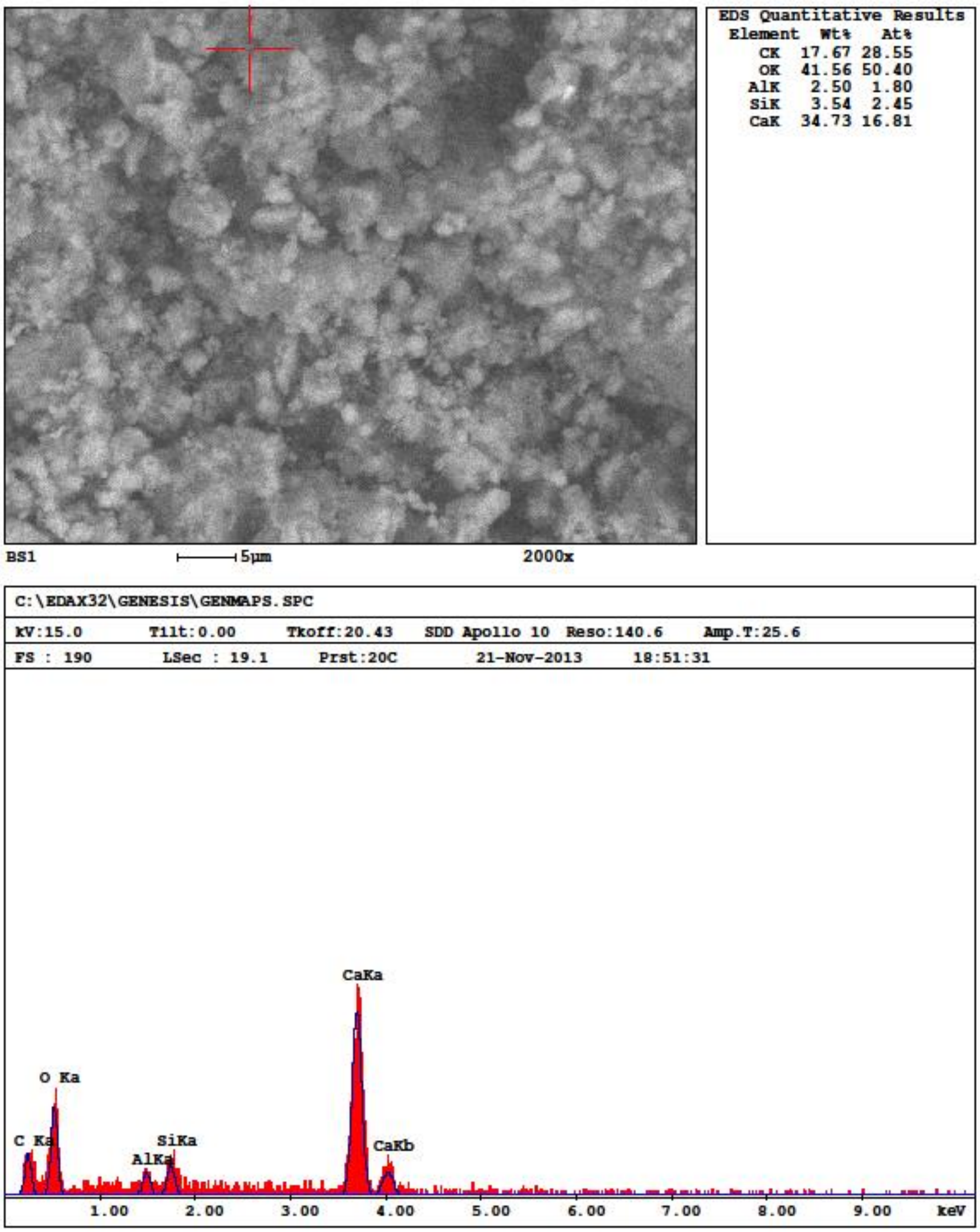
C:\EDAX32\GENESIS\GENMAPS.SPC

kV:15.0 Tilt:0.00 Tkoff:20.64 SDD Apollo 10 Reso:140.6 Amp.T:25.6

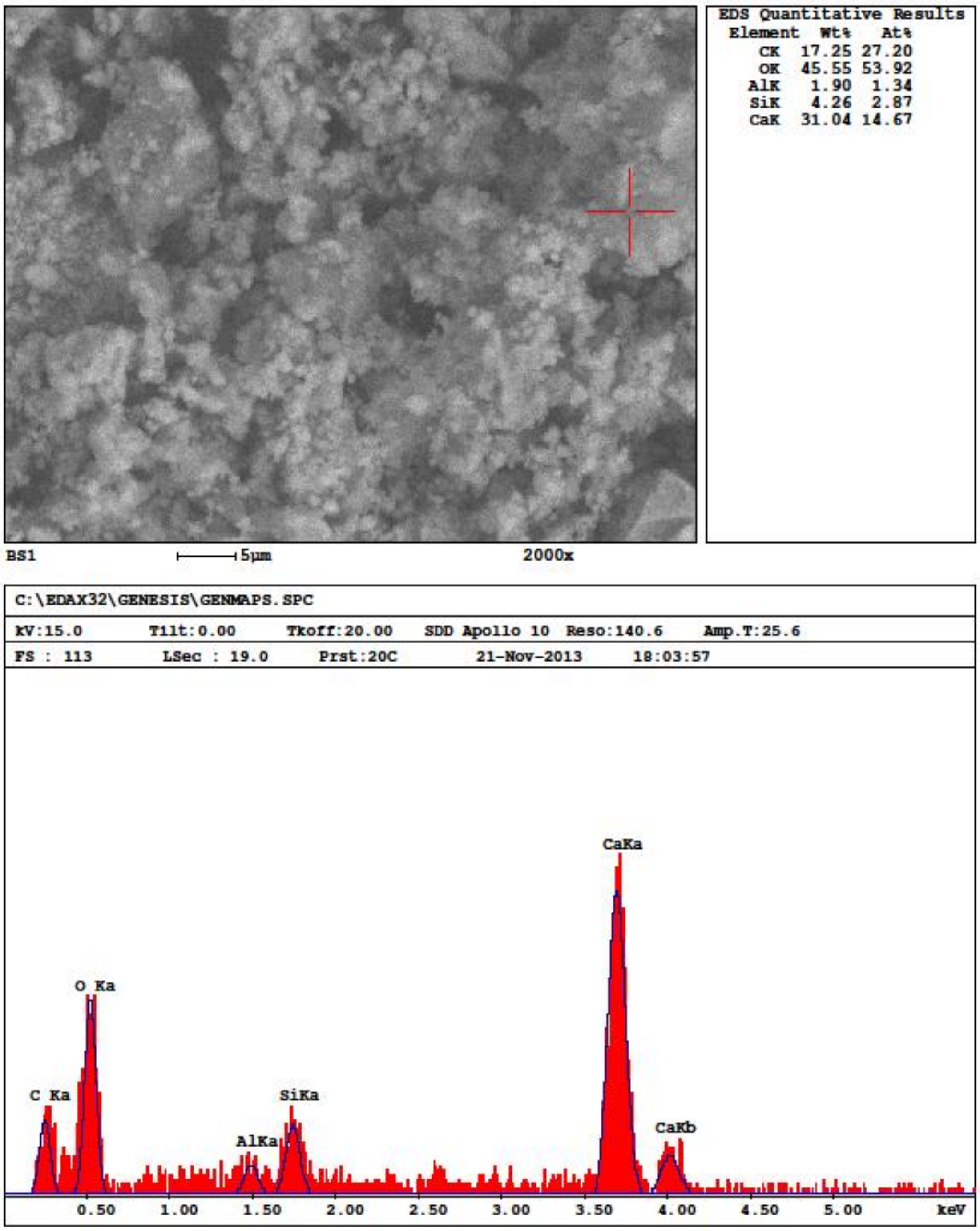
FS : 226 LSec : 18.9 Prst:20C 21-Nov-2013 18:35:30



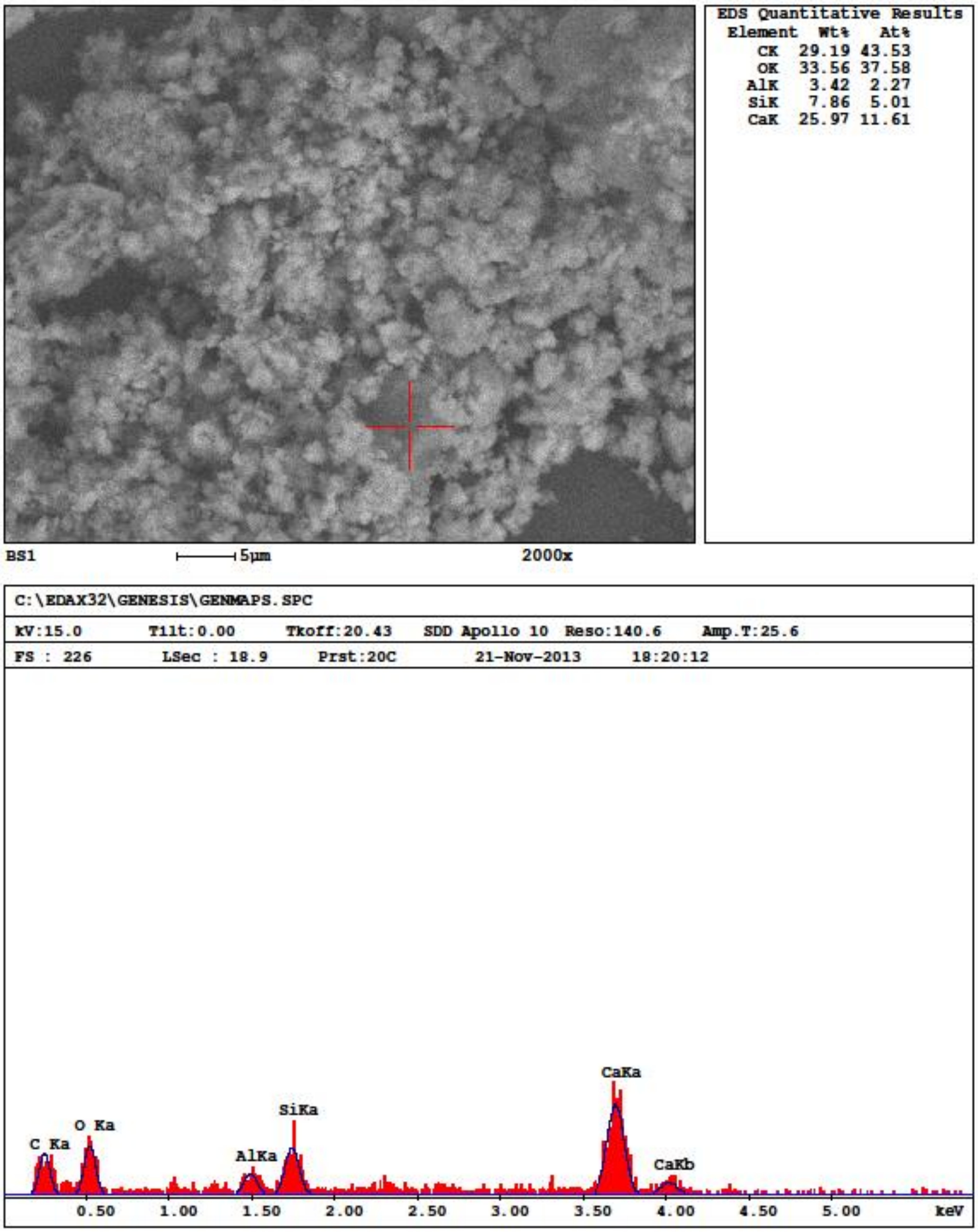
CE1-EDS Spectra:



SI1-EDS Spectra:

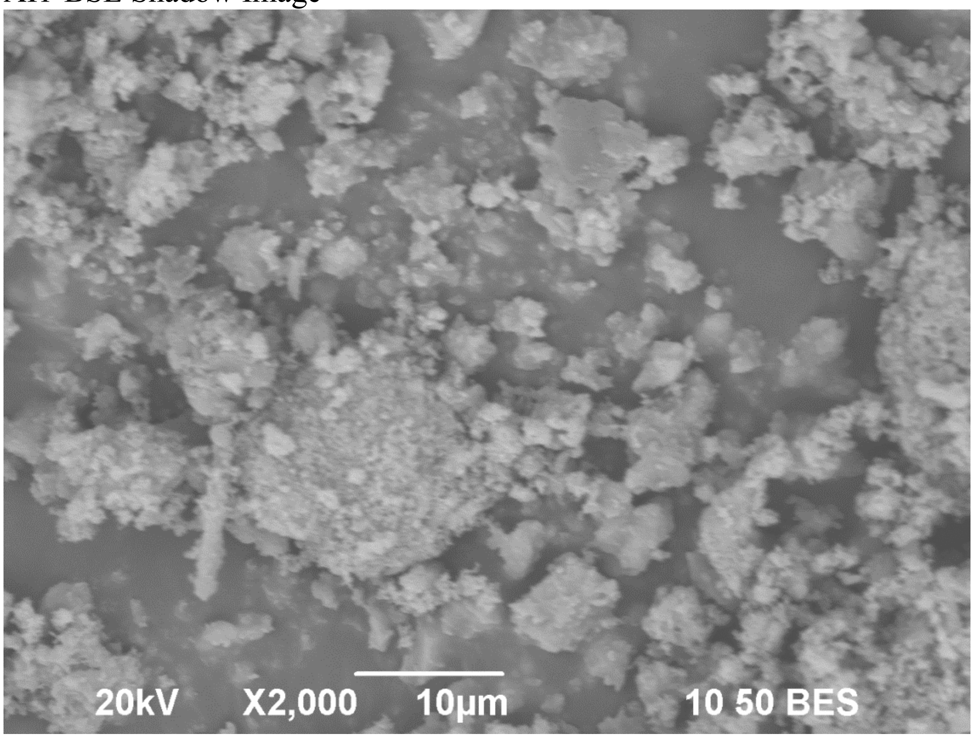


SI3-EDS Spectra:

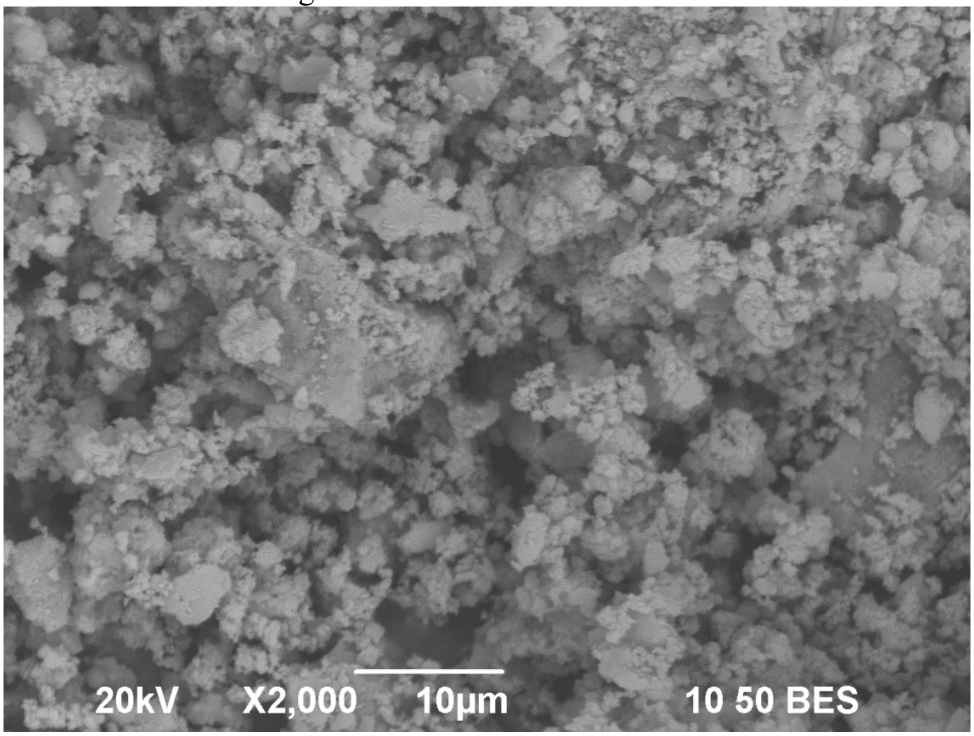


The following BSE images provide both compositional and topographical information. The brighter areas indicate elements with lower atomic numbers and energies. It is a visual representation of the EDS Spectra from above.

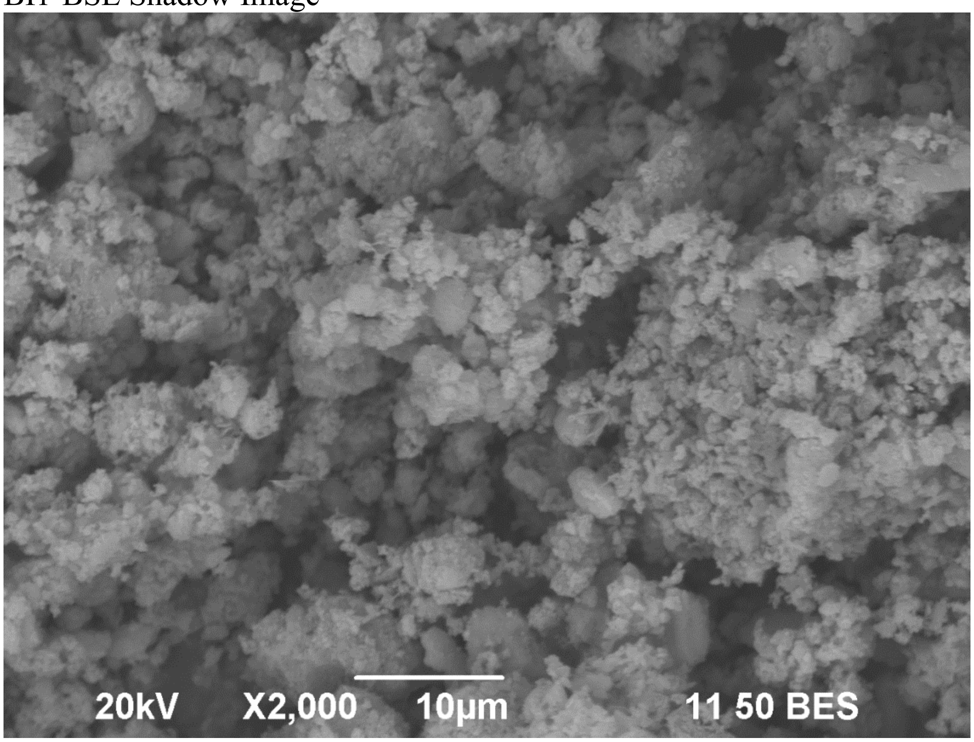
AI1-BSE Shadow Image



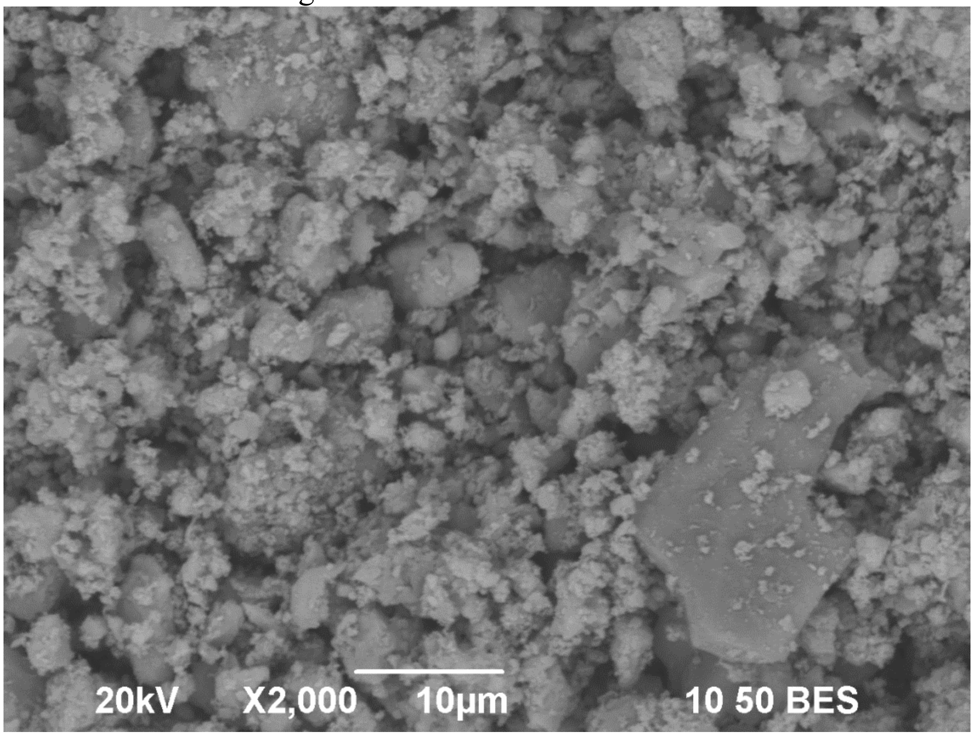
BE3-BSE Shadow Image



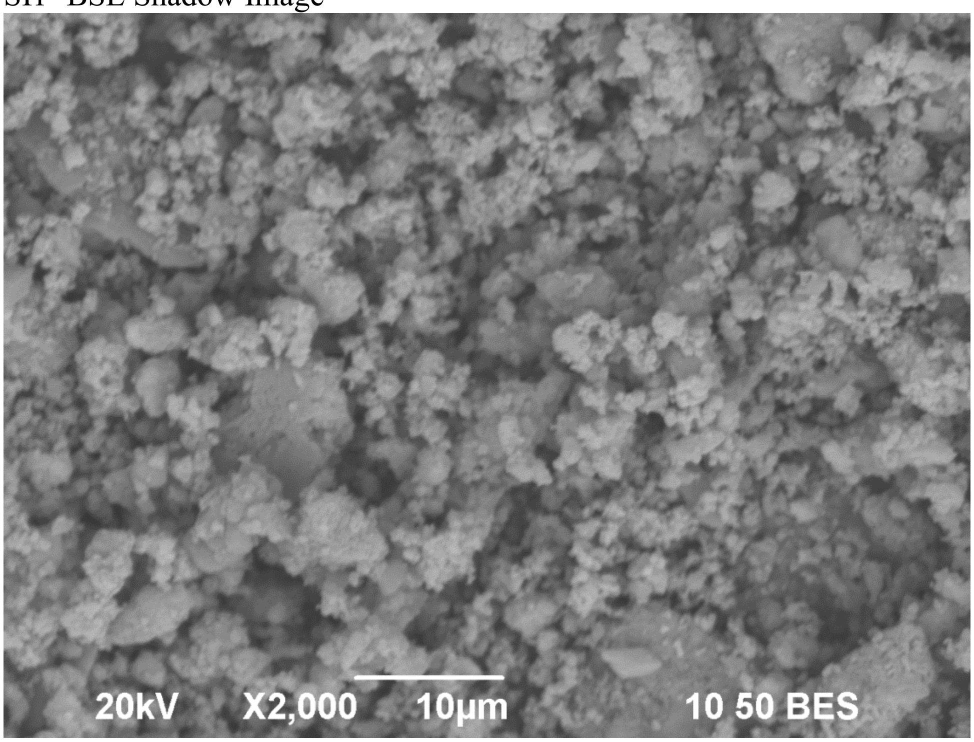
BI1-BSE Shadow Image



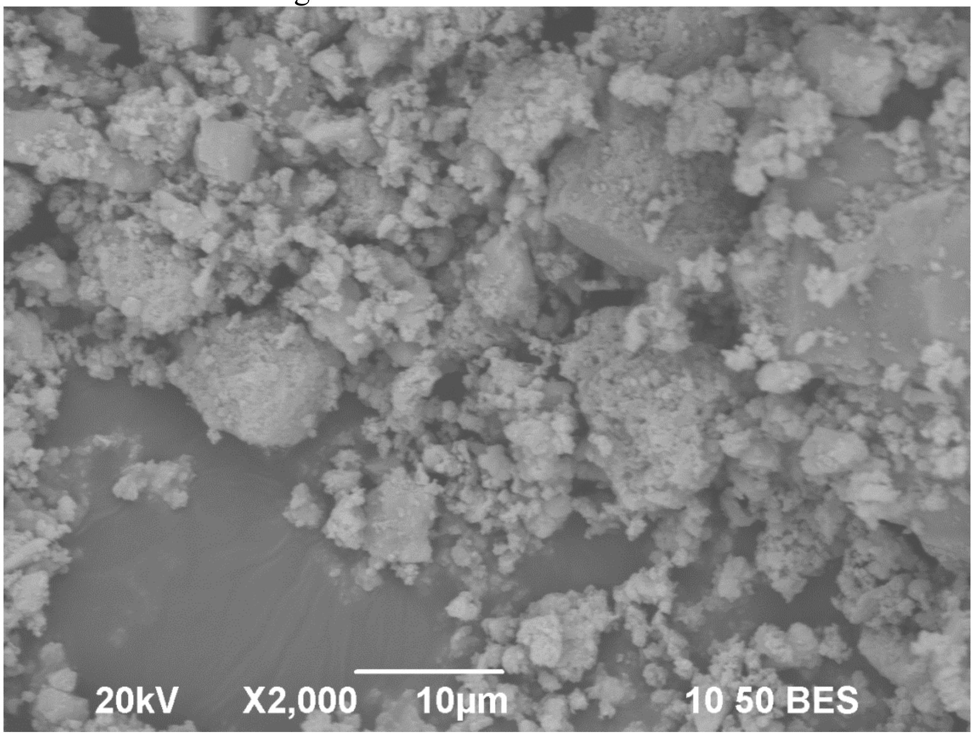
CE1-BSE Shadow Image



SI1- BSE Shadow Image



SI3- BSE Shadow Image



DISCUSSION OF RESULTS: Based on the SEM EDS spectra, the elements present (calcium, carbon, and oxygen) indicate that the composition of the samples is most likely calcium carbonate ($CaCO_3$) with small amounts of aluminum and silicon. These small amounts could indicate that clay is part of the binder as clay would include oxygen, aluminum and silicon in its chemical composition. Although the clay content has not been conclusively indicated using this technique, the EDS Spectra provides a good indication that clays might be present in the cement binder in trace amounts. Additional analysis with X-ray diffraction should aid in the identification of the chemical compounds within the samples.

Appendix F

LABORATORY REPORT: X-RAY DIFFRACTION ANALYSIS

DATE: February 4, 2014 and July 4, 2014
LABORATORY REPORT: Investigation with X-Ray Diffraction
PREPARED BY: Sarah B. Hunter

PURPOSE OF TESTING: This investigation uses x-ray diffraction (XRD) to determine the crystalline structure of the cement binder taken from several extant structures in Seguin, Texas. XRD analysis will identify the mineral structure and chemical compounds, providing both qualitative and quantitative data for the cement binder. This helps to determine whether clays were present in the cement binder. If clay is found in the binder, it will help assess the plausibility of each of the theories surrounding the concrete construction in Seguin. As learned from SEM EDS analysis, the samples all contain high percentages of carbon, calcium and oxygen. This serves as an indicator that the binder most likely contains calcium carbonate ($CaCO_3$) and possibly clay (aluminum and silicon).

SAMPLES: Please refer to Appendix "Sample Descriptions" for more information regarding samples. Several representative samples were selected based on preliminary investigations and extracted from each structure to be included in this portion of the study. The samples AI1, BE3, BI1, CE1, SI1, and SI3 were analyzed at the Jackson School of Geosciences. Two limestone samples from the pit located near the Moses Campbell property were analyzed at the Pickle Research Campus.

TEST METHOD: For this investigation, we used X-ray Diffraction (XRD). The work was completed in the University of Texas at Austin Jackson School of Geosciences E-beams Laboratory as part of the Fall 2013 Course "Analytical Methods Electron Microbeam Technologies" (GEO390R). We used the Electron Microbeam Laboratories standard operating procedure for the XRD. Additional XRD work was completed at the University of Texas at Austin Pickle Research Campus in the Construction Materials Research Group's Laboratory with the aid of Federico Aguayo.

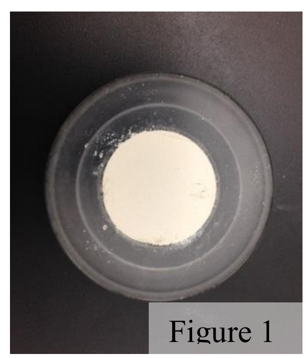


Figure 1

MATERIALS AND EQUIPMENT:
Sample Preparation: ceramic mortar and pestle, standard 3" diameter sieve set
Analysis: Bruker D8 Advance, specimen holder, glass slide

PROCEDURE:
Sample Preparation: The binder was isolated from the aggregate prior to analysis. Each sample was pulverized using a ceramic mortar and pestle, and then sieved using a standard sieve set.

Material collected in pan and sieve screens no. 100 & 200 were again pulverized, separated a second time in a standard sieve set, and material caught in the pan (less than 75 μm) was used for analysis. Once the samples were prepared, they were placed in the XRD specimen holders and leveled using a glass slide (fig. 1).

XRD Analysis: The uncoated samples were packed and leveled using a glass slide. The following experimental conditions were used for both analyses: Start position of 10° 2θ, Stop position of 80° 2θ, step size/increment 0.02°, time per step 0.5 seconds, generator voltage 40 kV, and generator current of 40 mA. The parameter file is on the following page. Based on the qualitative results from SEM with EDS, calcite, gypsum and quartz standards were selected as baselines for analysis.

File name: geo 390r_sarah.dql	**Print date:** 05-Dec-2013
Path: c:\diffdat1\sarah\geo 390r_sarah.dql	**Print time:** 09:34:53
	Program: XRD Wizard V2.9.0.22
	Page: 1/ 1

geo 390r_sarah.dql

Info

Wizard document type:	XRD
User:	Administrator
Site:	USA
Sample:	GEO 390R 2013_Sarah
Comments:	
User task file:	none
Total estimated time:	0:00:31:15 [d:hh:mm:ss]

Ranges

#1: Locked Coupled

Meas. delay time	0.00 [s]	= 0:00:00:00 [d:hh:mm:ss]
Estimated scan time	1875.00 [s]	= 0:00:31:15 [d:hh:mm:ss]
Scan mode	continous scan	
Start position	10	
Increment	0.019678	
#steps	3557	
Time per step	0.5 [s]	
Motorized slit changer:	OUT	
Synchronous rotation	ON	
Generator voltage	40 [kV]	
Generator current	40 [mA]	

Drives

Theta

Drive number	1
Encoder used	No
Position	5.0000 [°]
Oscillation	No

2Theta

Drive number	2
Encoder used	No
Position	10.0000 [°]
Oscillation	No

LynxEye

Electronic window	is current default

TEST RESULTS: The analysis indicated that calcite (92-93 wt %) and quartz (7 wt %) were present in all samples and a few samples presented gypsum in trace amounts. The following pages include the XRD diffractograms and the calcite, gypsum and quartz standards used for both analyses. Each of the diffractograms has the background removed and Ka2 stripped to aid in the peak identification.

Calcite Standard

Pattern : 00-005-0586 ***Radiation =*** 1.540600 ***Quality :*** High

$CaCO_3$

Calcium Carbonate
Calcite, syn

Lattice : Rhombohedral
S.G. : R-3c (167)
a = 4.98900
c = 17.06200
Z = 6

Mol. weight = 100.09
Volume [CD] = 367.78
Dx = 2.711
Dm = 2.710
I/Icor = 2.00

Additional Patterns: See PDF 01-072-1214 , 01-072-1937, 01-081-2027, 01-083-0577 and 01-083-0578. Analysis: Spectroscopic analysis: <0.1% Sr; <0.01% Ba; <0.001% Al, B, Cs, Cu, K, Mg, Na, Si, Sn; <0.0001% Ag, Cr, Fe, Li, Mn. Color: Colorless. General Comments: Additional weak reflections (indicated by brackets) were observed. Other form: aragonite. Pattern reviewed by Parks, J., McCarthy, G., North Dakota State Univ., Fargo, North Dakota, USA, ICDD Grant-in-Aid (1992). Agrees well with experimental and calculated patterns. Antacid. Sample Source or Locality: Sample from Mallinckrodt Chemical Works. Temperature of Data Collection: Pattern taken at 299 K. Unit Cell Data Source: Powder Diffraction.
Data collection flag: Ambient.

Swanson, Fuyat., Natl. Bur. Stand. (U.S.), Circ. 539, volume II, page 51 (1953)

CAS Number: 13397-26-7

Radiation : CuKα1
Lambda : 1.54050
SS/FOM : F30= 57(0.0158,33)

Filter : Beta
d-sp : Not given

d (Å)	I	h	k	l
3.86000	12	0	1	2
3.03500	100	1	0	4
2.84500	3	0	0	6
2.49500	14	1	1	0
2.28500	18	1	1	3
2.09500	18	2	0	2
1.92700	5	0	2	4
1.91300	17	0	1	8
1.87500	17	1	1	6
1.62600	4	2	1	1
1.60400	8	1	2	2
1.58700	2	1	0	10
1.52500	5	2	1	4
1.51800	4	2	0	8
1.51000	3	1	1	9
1.47300	2	1	2	5
1.44000	5	3	0	0
1.42200	3	0	0	12
1.35600	1	2	1	7
1.33900	2	0	2	10
1.29700	2	1	2	8
1.28400	1	3	0	6
1.24700	1	2	2	0
1.23500	2	1	1	12
1.18690	1	3	1	2
1.17950	3	2	1	10
1.17280	1	0	1	14
1.15380	3	1	3	4
1.14250	1	2	2	6
1.12440	1	1	2	11
1.06130	1	2	0	14
1.04730	3	4	0	4
1.04470	4	3	1	8
1.03520	2	1	0	
1.02340	1	2	1	13
1.01180	2	3	0	12
0.98950	1	3	2	1
0.98460	1	2	3	2
0.97820	1	1	3	10
0.97670	3	1	2	14
0.96550	2	3	2	4
0.96360	4	0	4	8
0.95620	1	0	2	
0.94290	2	4	1	0
0.93760	2	2	2	12

Quartz Standard

Pattern : 00-046-1045 ***Radiation =*** 1.540600 ***Quality :*** High

SiO_2

Silicon Oxide
Quartz, syn

Lattice : Hexagonal
S.G. : P3221 (154)
a = 4.91344
c = 5.40524
Z = 3

Mol. weight = 60.08
Volume [CD] = 113.01
Dx = 2.649
Dm = 2.660
I/Icor = 3.41

Additional Patterns: To replace 00-033-1161. See PDF 01-085-0335. Color: White. General Comments: Low temperature quartz. 2θ determination based on profile fit method. Temperature of Data Collection: Pattern taken at 296(1) K. Unit Cell Data Source: Powder Diffraction.
Data collection flag: Ambient.

Kern, A., Eysel, W., Mineralogisch-Petrograph. Inst., Univ. Heidelberg, Germany., ICDD Grant-in-Aid (1993)

Radiation : CuKα1
Lambda : 1.54060
SS/FOM : F30=558(0.0011,31)

Filter : Monochromator crystal
d-sp : Diffractometer

d (Å)	I	h	k	l
4.25499	16	1	0	0
3.34347	100	1	0	1
2.45687	9	1	1	0
2.28149	8	1	0	2
2.23613	4	1	1	1
2.12771	6	2	0	0
1.97986	4	2	0	1
1.81796	13	1	1	2
1.80174	1	0	0	3
1.67173	4	2	0	2
1.65919	2	1	0	3
1.60827	1	2	1	0
1.54153	9	2	1	1
1.45289	2	1	1	3
1.41841	1	3	0	0
1.38210	6	2	1	2
1.37496	7	2	0	3
1.37188	5	3	0	1
1.28791	2	1	0	4
1.25595	3	3	0	2
1.22832	1	2	2	0
1.19982	2	2	1	3
1.19779	1	2	2	1
1.18399	2	1	1	4
1.18017	2	3	1	0
1.15298	1	3	1	1
1.14065	1	2	0	4
1.11455	1	3	0	3
1.08155	2	3	1	2
1.06380	1	4	0	0
1.04772	1	1	0	5
1.04380	1	4	0	1
1.03461	1	2	1	4
1.01490	1	2	2	3
0.98958	1	1	1	5
0.98725	1	3	1	3
0.97834	1	3	0	4
0.97617	1	3	2	0
0.96078	1	3	2	1
0.92853	1	4	1	0
0.91816	1	3	2	2
0.91606	2	4	0	3
0.91518	2	4	1	1
0.90889	1	2	2	4
0.90085	1	0	0	6
0.89719	1	2	1	5
0.88891	1	3	1	4
0.88135	1	1	0	6
0.87817	1	4	1	2
0.85980	1	3	0	5
0.84584	1	1	1	6
0.84075	1	5	0	1
0.83592	1	4	0	4
0.82956	1	2	0	6
0.82539	2	4	1	3
0.81891	1	3	3	0
0.81168	3	5	0	2
0.80967	1	3	3	1

Gypsum Standard

Pattern : 00-033-0311 ***Radiation =*** 1.540600 ***Quality :*** High

$CaSO_4 \cdot 2H_2O$

Calcium Sulfate Hydrate
Gypsum, syn

Lattice : Base-centered monoclinic
S.G. : C2/c (15)

Mol. weight = 172.17
Volume [CD] = 495.37

a = 6.28450
b = 15.20790 ***beta =*** 114.09
c = 5.67760
a/b = 0.41324 ***Z =*** 4
c/b = 0.37333

Dx = 2.308
Dm = 2.320
I/Icor = 1.83

Additional Patterns: To replace 00-006-0046 and validated by calculated pattern 00-036-0432. See PDF 01-070-0982. Color: Colorless. General Comments: Preferred orientation enhances 0k0 reflections. Sample Preparation: Sample prepared by adding "H2 S O4" to a "Ca (N O3)2" solution; the precipitate was filtered out, washed in water and bottled while moist; the crystals were dried immediately before use with care taken to prevent dehydration. Temperature of Data Collection: Pattern taken at 298 K.
Data collection flag: Ambient.

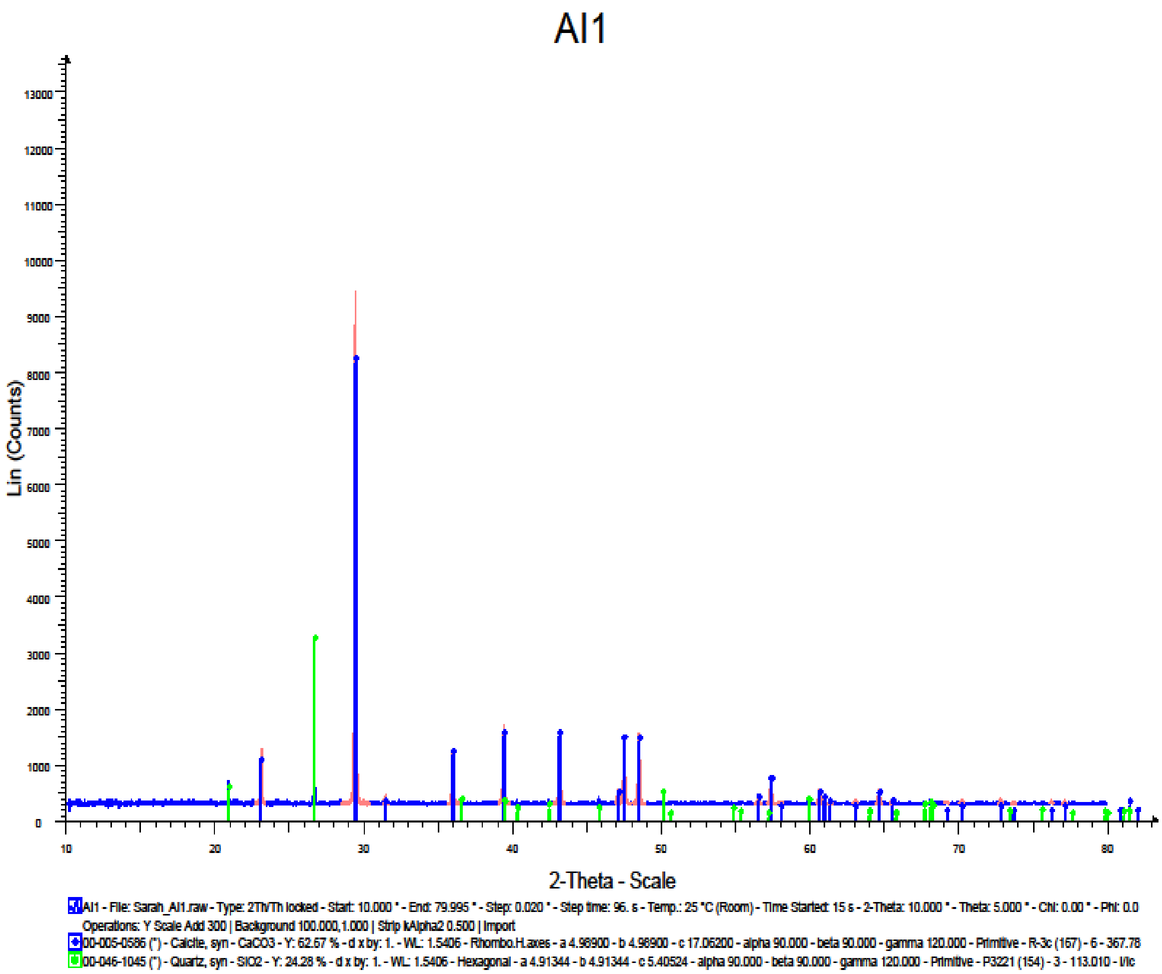
Natl. Bur. Stand. (U.S.) Monogr. 25, volume 17, page 16 (1980)

Radiation : CuKα1
Lambda : 1.54060
SS/FOM : F30= 52(0.0117,49)

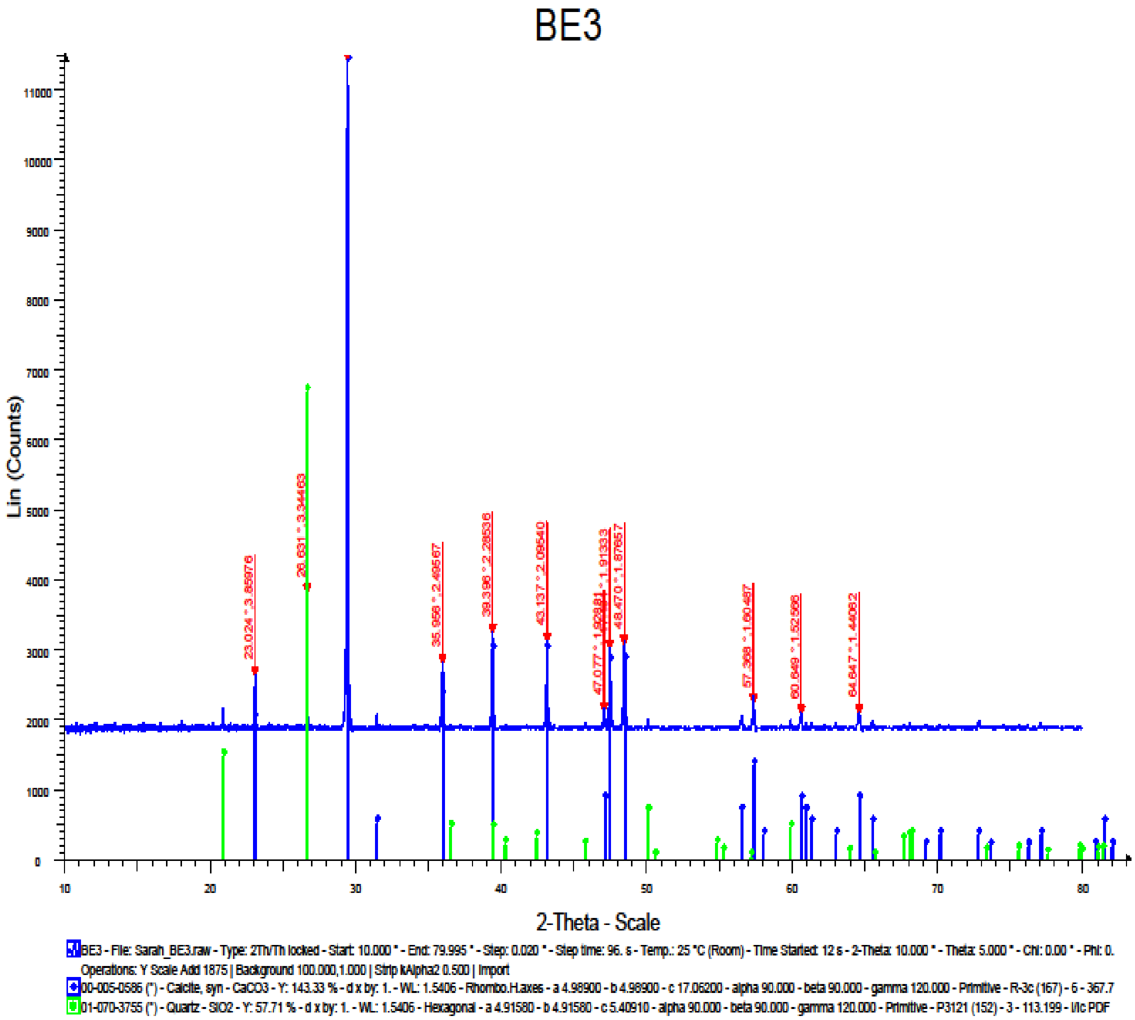
Filter : Monochromator crystal
d-sp : Diffractometer

d (Å)	*I*	*h*	*k*	*l*
7.63000	100	0	2	0
4.28300	100	0	2	1
3.79900	17	1	3	0
*3.79900	17	0	4	0
3.17200	4	1	1	1
3.06500	75	0	4	1
2.87300	45	-2	2	1
2.78900	10	-1	1	2
2.73200	2	1	3	1
2.68500	35	1	5	0
*2.68500	35	2	2	0
2.59700	6	-1	5	1
2.53400	2	0	6	0
2.49500	11	-2	0	2
2.47600	1	-1	3	2
2.45200	6	0	2	2
2.40600	4	-2	4	1
2.29100	1	2	4	0
2.21900	15	1	5	1
2.14200	2	0	4	2
2.08600	25	-2	4	2
2.07400	15	-3	1	1
*2.07400	15	-1	5	2
2.04800	6	1	1	2
2.03200	1	1	7	0
1.99200	4	-1	7	1
1.96300	3	-2	6	1
1.89980	16	0	8	0
*1.89980	16	2	6	0
1.87950	12	2	4	1
1.86500	3	-1	1	3
1.81180	13	0	6	2
1.79950	6	-2	2	3
1.78440	9	0	8	1
1.77850	12	-2	6	2
1.70930	1	1	5	2
1.68460	3	0	2	3
1.66400	6	-2	4	3
1.64560	4	2	6	1
1.62090	9	1	9	0
*1.62090	9	-2	8	1
1.60050	1	-1	9	1
1.58460	4	2	8	0
1.53270	2	0	8	2
1.52090	1	-4	2	2
*1.52090	1	0	10	0
1.51190	1	-2	8	2
1.49820	1	1	9	1
1.49470	1	-2	6	3
1.45910	3	0	10	1
*1.45910	3	-3	7	2
1.43920	5	-4	4	1
1.43540	3	3	7	0
1.42780	2	0	6	3
*1.42780	2	2	8	1
1.41780	3	-2	0	4
1.40150	2	-4	2	3
1.36570	5	-2	10	1
*1.36570	5	2	6	2
1.34400	1	2	10	0
*1.34400	1	1	11	0
1.33240	2	-1	11	1
1.32620	4	-2	8	3
1.32340	4	-4	6	2
1.27850	1	0	8	3
1.27220	1	1	11	1
1.26740	1	0	12	0
1.24810	3	-4	0	4
*1.24810	3	4	6	0
1.24410	2	2	10	1
1.23360	3	2	8	2
1.23090	2	0	12	1
*1.23090	2	-4	2	4

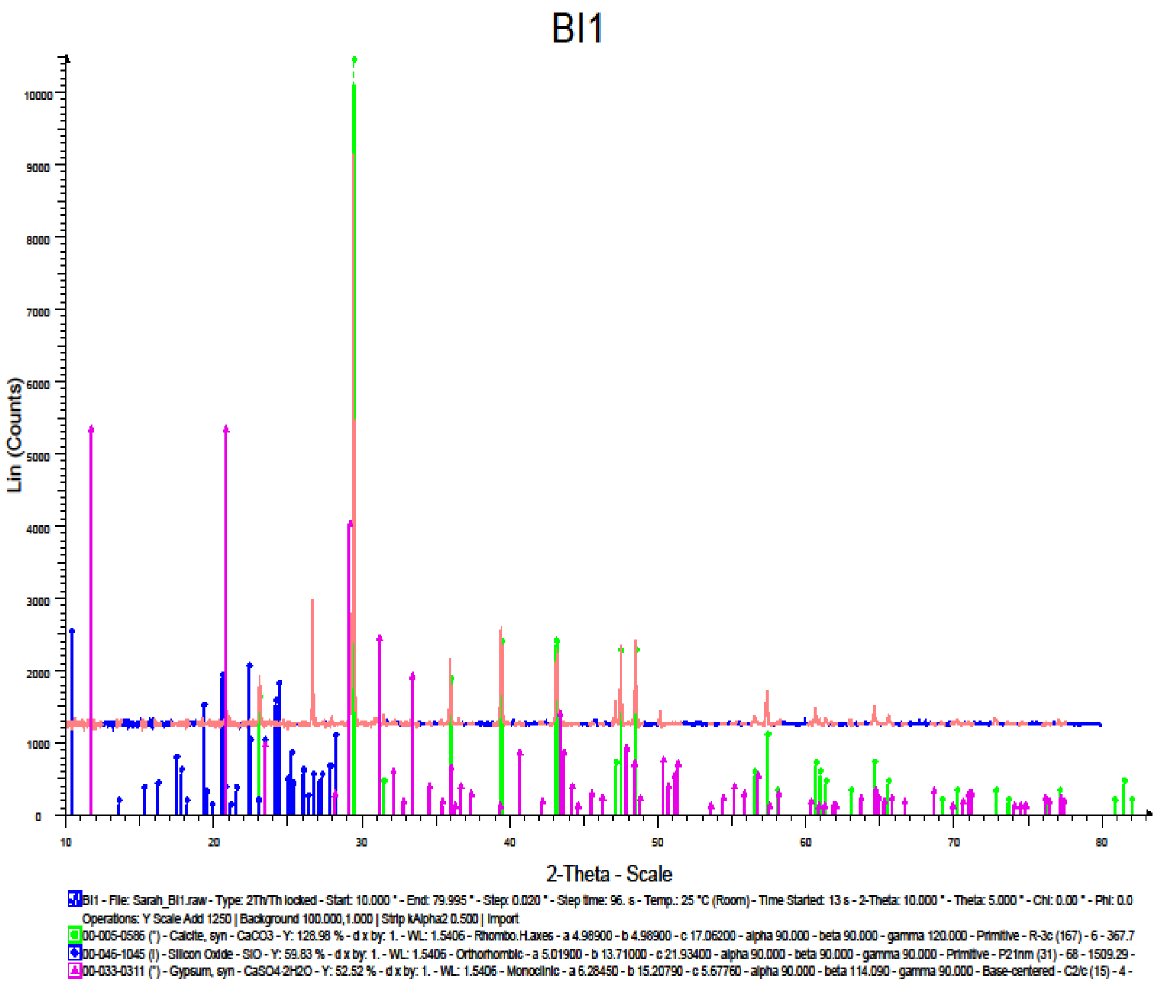
AI1 Diffractogram



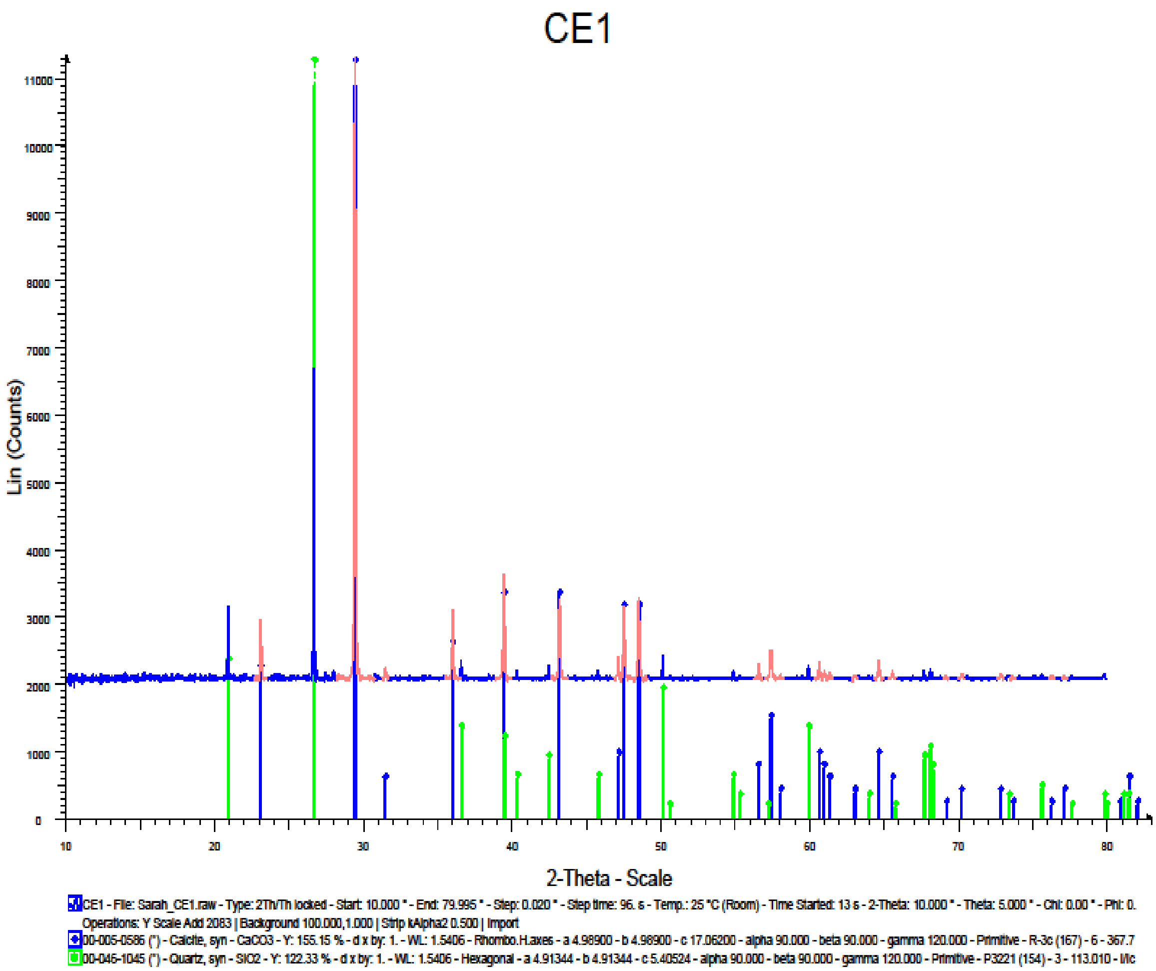
BE3 Diffractogram



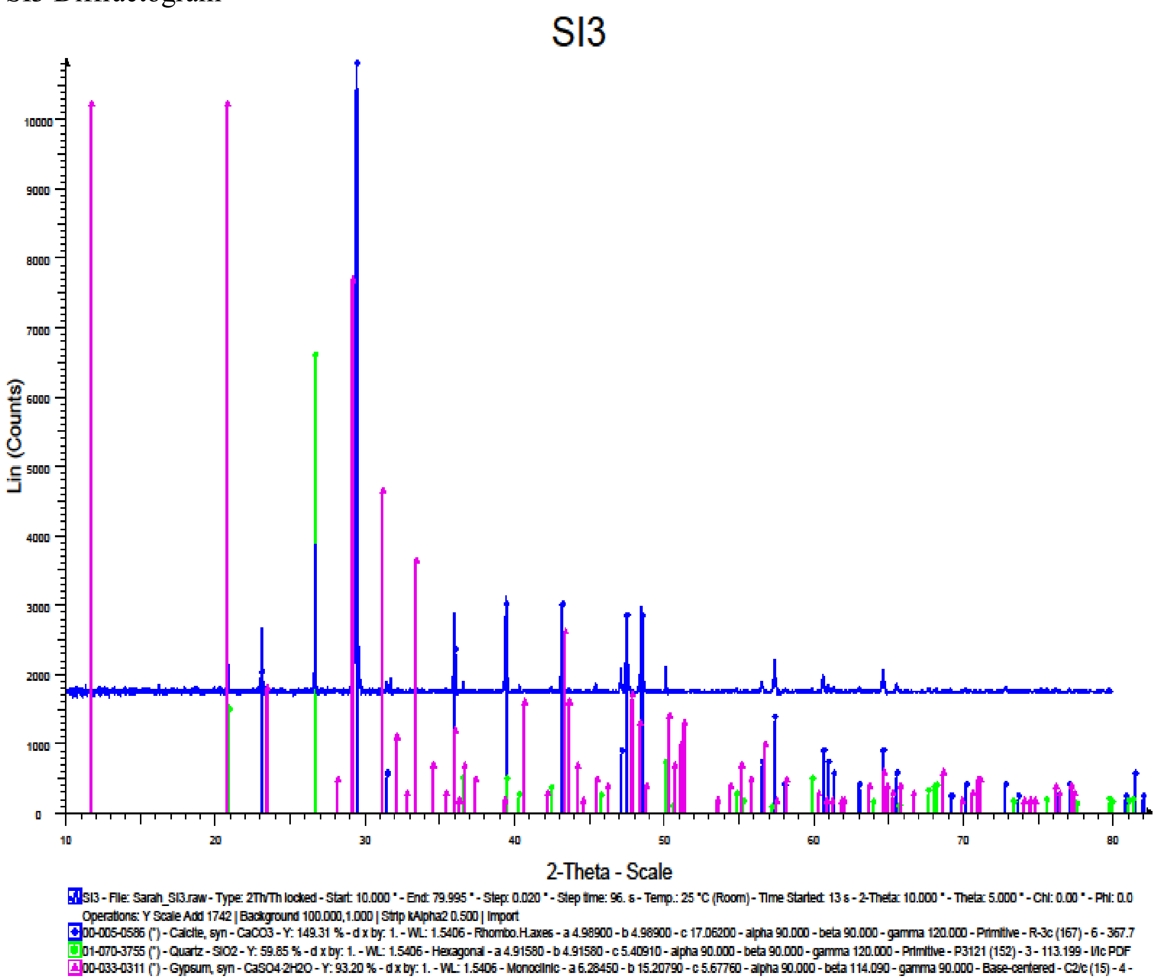
BI1 Diffractogram



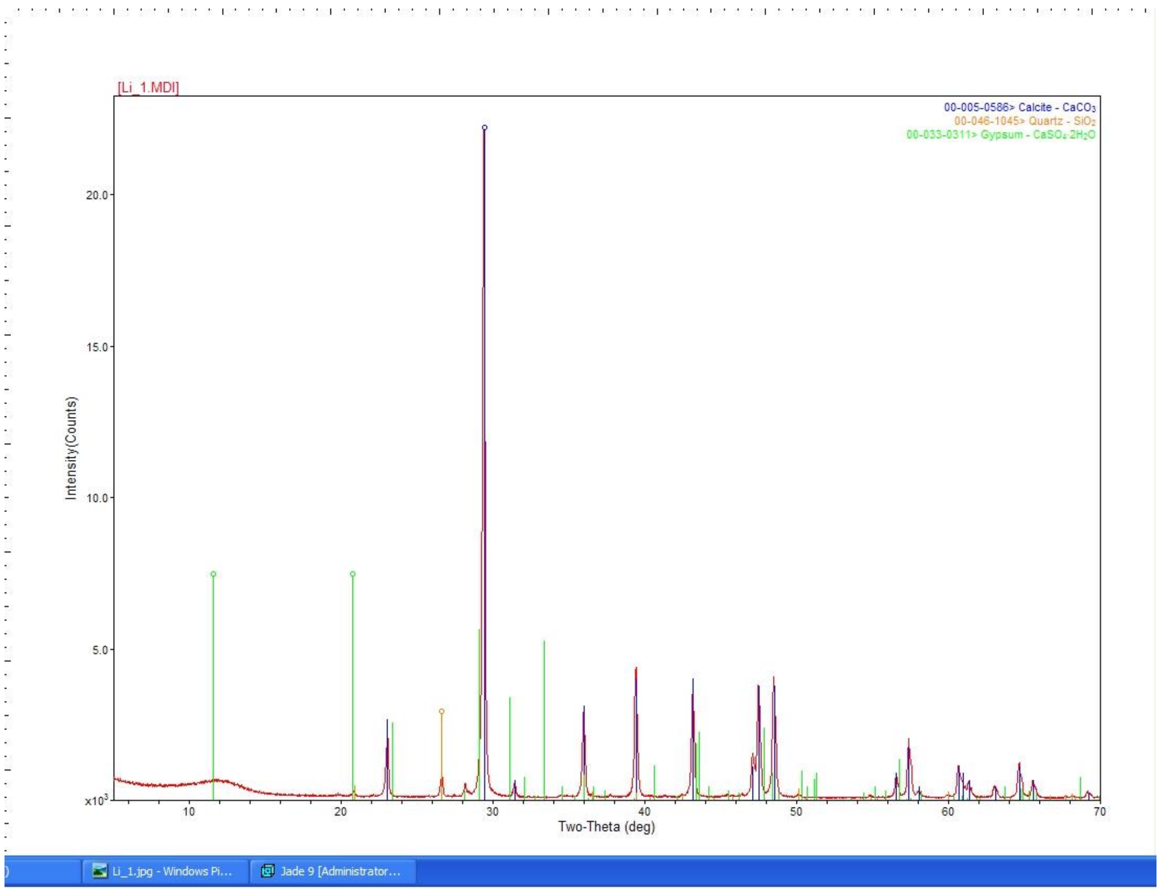
CE1 Diffractogram



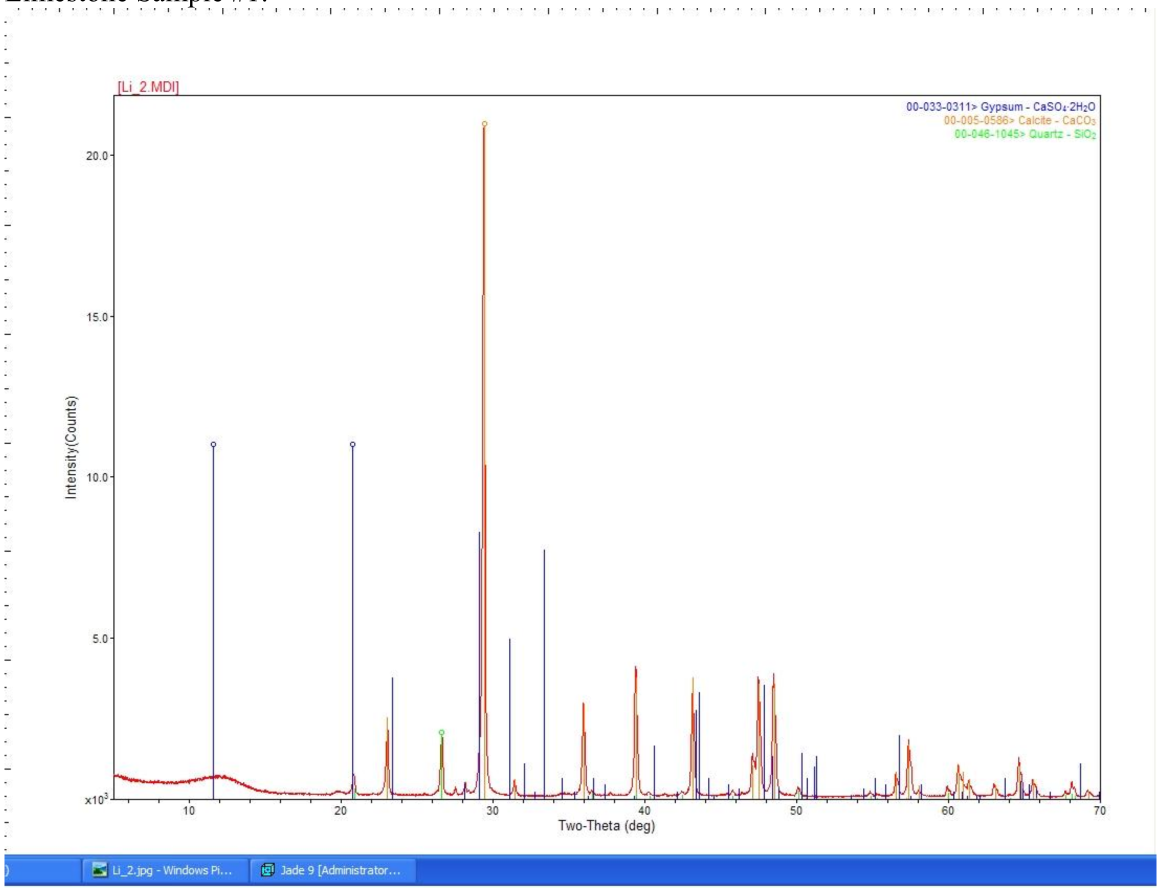
SI3 Diffractogram



Limestone Sample #1:



Limestone Sample #1:



DISCUSSION OF RESULTS:

Based on the SEM EDS spectra the elements present indicate that the composition of the samples is calcium carbonate ($CaCO_3$) (or calcite) with trace amounts of aluminum and silicon. These trace amounts of these elements could be from a number of sources such as other impurities in the limestone or added clay content. The calcium carbonate is confirmed with XRD analysis, but the clay content in under the detectable limit. Also, during calcination, kaolinite's crystal structure collapses and becomes an amorphous metakaolinite.[47] Finding koalinite's chemical compound with XRD would not be possible if it had been fired. The calcium aluminum silicates or silicate hydrates that would indicate the presence of clays were either below the detection limit of the XRD or not present in the sample.

The two limestone samples had diffractograms that were similar to the binder material. Both samples were calcite (calcium carbonate) with trace amounts of gypsum and quartz.

When using these two analytical methods together, it can be seen that the representative samples have similar compositional percentages and materials. With further comparison to the limestone samples, it is very possible that the same construction method was being used for all of the structures using materials from similar sources over a period of time.

[47] A. Elena Charola and Fernando M.A. Henriques, "Hydraulicity in Lime Mortars Revisited," in *PRO 12: International RILEM Workshop on Historic Mortars: Characteristics and Tests*, ed. Peter Bartos, Caspar Groot, and John J. Hughes (RILEM Publications SARL, 1999), 97-99.

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