# A MINERALOGICAL AND RESOURCE PROCUREMENT STUDY OF PRE-CIVIL WAR FORT CONSTRUCTION USING STONE AND MORTAR IN OKLAHOMA

By

# PATRICK DEE HURLEY

Bachelor of Science in Horticulture

Oklahoma State University

Stillwater, OK

2008

Submitted to the Faculty of the Graduate College of the Oklahoma State University in partial fulfillment of the requirements for the Degree of MASTER OF SCIENCE May, 2013

# A MINERALOGICAL AND RESOURCE PROCUREMENT STUDY OF PRE-CIVIL WAR FORT CONSTRUCTION USING STONE AND MORTAR IN OKLAHOMA

Thesis Approved:

Dr. Brian J. Carter

Thesis Adviser

Dr. James O. Puckette

Dr. Leland C. Bement

#### ACKNOWLEDGEMENTS

I would like to thank Dr. Brian Carter for accepting me into this program and giving me the opportunity to achieve this level of academic excellence and continuing to further my understanding of natural science from a historical and scientific point of view. I would also like to thank Dr. James Puckette for his help and expertise in geology and data interpretation. Additionally, my thanks to Dr. Leland Bement, for taking the time and for sharing his expertise in archeology, as well as, his vast knowledge of the forts and their shared history. I am also very grateful for the help and assistance of Scott Fine who went above and beyond in my moment of need. I would like to extend a special thank you to my immediate supervisor Dustin MacKenzie. If he had not given me time off from work, earning this degree would have been exponentially more difficult.

I would like to thank my family and friends for their continued encouragement and prayers over the last five years for the completion of this degree. However, I would like to thank my mother, Karen, most of all. Without her continued love, support, and enduring spirit I doubt I would have had the patience or discipline needed to continue along this arduous path. She has continued to believe in me when others could not or would not see my potential. It is through her unwavering strength, character, and personal courage, as well as our shared belief in the Almighty, that I have been this successful in life. For this reason, I am forever blessed, honored, and proud to call her my mother.

#### Name: PATRICK DEE HURLEY

#### Date of Degree: MAY, 2013

# Title of Study: A MINERALOGICAL AND RESOURCE PROCUREMENT STUDY OF PRE-CIVIL WAR FORT CONSTRUCTION USING STONE AND MORTAR IN OKLAHOMA

#### Major Field: MASTER OF SCIENCE

Abstract: Much is known about the arduous lifestyles of the early nineteenth century United States soldier. However, little is known of the actual laborious fort construction methods these soldiers used as they upgraded from lumber-only to stone-mortar and lumber structures along the western frontier. Specifically, little is known about the mortar production from; natural limestone formations, the process of mortar-stone construction, and of the possible sources for these raw materials. In order to identify how soldiers and workmen produced these mortars, mortar samples were taken from Forts Gibson, Towson, and Washita in eastern Oklahoma. These forts were constructed in 1824, 1824 again in 1831, and 1840 respectively. Each mortar sample was analyzed using thin section analysis, x-ray diffraction (XRD) analysis; calcium carbonate equivalents (CCE) analysis, and total inorganic carbon content. Historical documents were also used to find evidence of possible mortar-production procedures or locations for raw materials. Results indicated that the limestones used in the mortar production were selected based on their natural impurities, most likely for high silica content. The calcium carbonate and silica content were used to ascertain the best mortar composition when producing mortar at or near the fort. Workers needed limestone that yielded a minimum of 60% CCE. Dolomitic limestone would be preferable over pure calcitic limestone. However, Forts Gibson and Towson contained magnesium calcite. Magnesium calcite is stronger than pure calcite, thus more durable and suitable for construction. Thin section analysis showed particle size and ratios for grain, mortar, and void space. Each fort had a mean relationship of 50% grain, 43% mortar, and 7% void space. The grain size used for mortar production measured between .35 and .39 mm. Of those grains, 93.72% were identified as quartz (sand). Though no formal recipe was found while conducting this research, it is possible to re-contruct a period-like mortar from local materials and period methods.

# TABLE OF CONTENTS

Chapter	Page
I. INTRODUCTION	1
Introduction	1
Brief Background of Cement	1
Objectives	3
Justification	5
Hypotheses	6
II. REVIEW OF LITERATURE	7
Historical Background	7
Examples of Early Structures and Materials	
Prominent Historical Architecture	9
Historical Milestones Mortar Development	10
Natural Cement Classification and Process Section	10
Scientific Techniques and Testing	
Modern Testing	15
Thin Section Testing	
Limestone Classification	10
Calcite	18
Dolomite	18
X-Ray Diffraction	
III. MATERIALS AND METHODS	20
Sampling Procedures	20
Fort Location at Geologic Setting	21

Sampling Procedures	20
Fort Location at Geologic Setting	21
Fort Gibson	
Fort Washita and Towson	
Thin-Section Analysis	
Description of General Thin-Section Process	
Photographing Thin-Sections	
Count Parameters	

# Calcium Carbonate Equivalent Measurements32Modified Pressure Calcimeter Test32Calcium Carbonate Equivalent by Titration34Historical Documentation34IV. RESULTS AND DISCUSSION36Source Limestones for Fort Mortar36Calcium Carbonate Equivalents36X-Ray Diffraction38Thin-section Analyses of Mortar42Grain Counts- Fabric Analysis42Grain Counts- Identification and Size42Historical Documentation45Fort Washita45Fort Gibson46Fort Towson47

Chapter

V. SUMMARY	49
REFERENCES	53

# Page

# LIST OF TABLES

Table	Page
1: Sample identification, fort and description	21
2: Calcium carbonate equivalents for limestone	37
3: Fabric analysis: point counts	42
4: Comparison between quartz and non-quartz	43
5: Percent major non-quartz minerals and rocks	44

# LIST OF FIGURES

Figure	Page
1: Physical location of forts	4
2: Geologic map of fort locations	23
3: Footer for original wall at Fort Gibson	24
4: Excavations during 2008 field season	24
5: Original barracks at Fort Washita	25
6: Interior of barracks after 1st reconstruction	25
7: Limestone wall where 1st generation mortar sampled	26
8: Multi-generational mortars in wall	26
9: Foundation of post hospital at Fort Washita	27
10: Top view of kiln at Fort Towson	27
11: Interior of kiln at Fort Towson	28
12: Original wall of officer's quarters	28
13: Partially re-constructed wall at Fort Towson	29
14: Piece of 1st generation mortar at Fort Towson	29
15: Comparison of thin sections	31
16: Fort Gibson x-ray diffraction	39
17: Fort Washita x-ray diffraction	40
18: Fort Towson x-ray diffraction	41

19: Sand distribution curve45
-------------------------------

#### CHAPTER I

#### INTRODUCTION

People in societies that pre-date the written word built structures to honor their cities, gods, and countries. Since people began using architecture and construction methods they have looked for ways to fortify and make their structures fixed and impervious to time, natural elements, and conquering nations. Some of these structures, ones made with exceptional quality lasted throughout the centuries. This paper focuses on how military personnel in the early to middle 19<sup>th</sup> century produced materials and built structures necessary to serve and function as fortifications on the United States frontier of "Indian Territory." Fortifying an area on an open prairie is difficult from a strategic perspective because there is little-to-no cover or concealment. It is a vast, open space; sometimes having rolling hills and other times is flat. These forts were erected in the most strategic locations possible. These areas provided fresh water for consumption and transportation (major river system), cover (tree lines), and source for raw materials, such as stone and mortar. Historically and logistically fort locations must provide these criteria.

#### Brief Background of Cements

Architects and builders have used binding agents to solidify their creations for more than two millennia. Cements and mortars were used to bind mankind's structures dating back to the first pyramids in Egypt. The Egyptians used mud or clay initially, to build their pyramids (McKee, 1973). They also discovered that gypsum-lined beds allowed for the easier removal of stones to put into position. Additionally, the Egyptians added lime to their mortar circa 4000 B.C. as it was found to increase rigidity and rate at which the mortar hardened (Boynton, 1980). The discovery of "natural cements" is credited to the Romans who first noticed that when they added a volcanic rock to the cement mixture predominately found around Mt. Vesuvius that it would set up under water. This type of cement became the dominant variety for the engineering accomplishments constructed in or around bodies of water, like the aqueducts. Clarification of key terms used for closely related construction materials are needed as follows:

- Cement- a finely pulverized powder of alumina, silica, lime, iron oxide, and magnesium oxide burned together in a kiln and used as an ingredient of mortar and concrete,
- Mortar- a plastic building material (as a mixture of cement, lime, or gypsum plaster with sand and water) that hardens and is used in masonry or plastering, and
- c. Concrete- a mass formed by concretion or coalescence of separate particles of matter in one body; a hard strong building material made by mixing a cementing material (such as Portland cement which is made by "burning to incipient fusion a finely ground artificial mixture, consisting essentially of lime, silica, alumina, and some iron oxides...Ries and Watson, 1936) and a mineral aggregate (such as sand and gravel) with sufficient water to cause the cement to set and bind the entire mass.

Mortars were the beginning of man's building technology, which bound stones or other construction agents. Mortar is simple and cost efficient compared to cement. Cements evolved out of man's constant search for better and stronger methods to build larger and grander structures. Cements require the addition of materials that allow it to set stronger and faster compared to mortar. Cements also require a kilning process for limestone and small granule size, created by a sieving process. Modern concrete is merely aggregates, (usually sand to gravel size elements) mixed with Portland cement and water.

#### Objectives

The objective of this project is to understand and produce finite evidence of how United States frontier forts, circa 1830-1870, were constructed and if it was done with local or outsourced resources. This paper will focus on using calcium carbonate equivalents (CCE); thin section analysis, historical documentation, and x-ray diffraction analysis to delineate how and with what materials these frontier outposts were constructed:

1. Calcium carbonate equivalents (CCE)- these equivalents were measured to determine if the samples taken meet the minimal requirement for construction purposes. In other words, if the local limestone samples did not meet a minimum requirement of 60%, or greater CCE (Varas, 2005), then it may be inferred that lime used in construction was brought in from another source,

2. thin section analysis-

a. grain counts- samples of the mortar from Forts Gibson, Wichita, and Towson, Oklahoma were acquired (Figure 1). Analysis of the mortar in the form of thin sections will allow identification of the dominant minerals and identified a possible local sand source for these samples. These thin sections allow a count and percentage of mineral content will be used to determine if these samples came from local areas by comparing ratios of minerals and rock constituents.

3



Figure 1. Physical location of Oklahoma forts included in this study.

b. fabric analysis- taken from part of the thin section analysis and using a polarizing microscope, the slides are used to determine a ratio of grain: mortar: pore space (empty spots in the slide). This analysis helped determine a possible "recipe" for mortar used to construct these forts,

3. historical documentation- records from the United States National Archives involving materials used for construction and a possible recipe for the mortar will be reviewed, and

4. x-ray diffraction (XRD)- A technique, used in tandem with thin section analysis. This method exposes the samples to CuK alpha radiation and allows the mineralogical composition to be determined by analyzing the intensity of the mineral refraction (Riccardi, 1997). Using the samples collected from the sites, the limestone was ground in a ball mill and placed in a metal

pocket slide and submitted to the x-ray scan. As outlined by Schnabel (2009), x-ray diffraction is a technique used on powdered samples that enables the identification of minerals.

#### Justification

Initially, the reason for undertaking this project was to understand how these pre-civil war forts in Oklahoma were erected from locally derived source materials. In other words, answering how these forts were built, what materials were used, and location from where these materials were utilized. However, there is no evidence of kilns at Forts Gibson and Washita. It may be possible that some of these materials did not come from local sites and were transported to the forts by boat or wagon. Second, this paper focuses on understanding how and with what materials these forts were constructed because, from a reconstruction perspective, it is important to maintain and preserve these structures for future generations. Also, it is of key importance to restore and preserve these historic monuments with period materials to the best of our knowledge and understanding. Repairing historic or ancient constructions is not a "new" concept. In fact, most buildings of interest, like the Roman coliseum, aqueducts, and castles, are all highly sought after to restore to their once grand status. The Forts of Gibson, Washita, and Towson are no different than any other historic monument in need of preservation. The problem arises when repairs are needed, but no economical method exists to repair them with materials from the period. For example, the forts, over the years, have needed patchwork on the mortar to keep them structurally sound. There is currently no economic or local method, shy of using Quikrete® Portland cement, to repair these mortars. A process and methodology from the era is needed to keep these monuments in their original condition for future generations to enjoy.

#### Hypotheses

This paper strives to test the following hypotheses:

1. The null hypothesis: The selected Oklahoma frontier forts were constructed by manual labor with materials (i.e. lumber, mortar, limestone, sand) provided and retrieved from local sites. The assumption is the people at these forts were using and making natural cement. This hypothesis seems to be historically supported with the fact that the first commercial Portland cement in the United States did not come into existence until 1871. Thus, these cements and mortars pre-dated Portland by thirty to forty years,

2. A secondary hypothesis is: Materials for mortar production were shipped in via the local river system, including out-sourced, contracted labor, with appropriate skill set to produce mortar from local resources,

3. A tertiary hypothesis is: These forts were constructed with a combination of the both local and out-sourced or contracted materials and labor for the best possible outcome.

#### CHAPTER II

#### **REVIEW OF LITERATURE**

#### Historical Background

#### Examples of Early Structures and Materials

The modern cement or mortar after about 1871, as used first by John Smeaton in England, is directly related to cement discovered by the Romans in Europe, specifically, the Roman architect Vitruvius. Elliott (1992)paraphrases Vitruvius when he writes, "Limestone when taken out of the kiln...is found to have lost about a third of its weight owing to the boiling out of the water. Therefore, its pores being thus opened and its texture rendered loose, it readily mixes with sand, and hence the two materials cohere as they dry, unite with the rubble, and make a solid structure...There is also a kind of powder which from natural causes produces astonishing results. It is found in the neighborhood of Baiae and in the country belonging to the towns round about Mt. Vesuvius. This substance, when mixed with lime and rubble, not only lends strength to buildings of other kinds, but even when piers of it are constructed in the sea, they set hard under water" (Elliott, 1992). Similarly, in 1755 the Eddystone Lighthouse located fourteen miles southwest of Plymouth Harbor, in England burned down. A young engineer named John Smeaton was tasked with its reconstruction. He set out to choose cement that would be strong and cost effective...Smeaton wanted a pure limestone but was the first to notice that burning the impure limestone was "wasteful" and he was the first to say that the strength to which the cement hardens is not directly related to strength of the parent rock. According to Elliot (1992), this was the prevailing concept of cement dating back to Vitruvius.

Industrialization is often thought to have taken place during the turn of the 20<sup>th</sup> century (1880-1900). However, the first European settlers in the United State were producing goods to be exported. For example, less than two years after Jamestown was founded, the settlers were making glass, pitch, and tar, soap ashes, and lumber products. In fact, in 1608, a group of Dutch settlers constructed a glass house using "local sand (with lime and potash) (Gordon and Malone, 1994)." In 1790, when the Middlesex Canal was constructed, imported hydraulic cement was used. However, in 1820, while building the Erie Canal, local cement stone was pulled from Chittenango, New York.

The most recent of the construction mediums is that of reinforced concrete. Beginning in the late 1800's, architects were discovering that, by adding steel reinforcements, they could add strength to their concrete structures. Ernest Ransome is attributed to being the first to show the conceivable uses for reinforced concrete structures in the 1880's. Ransome's best-known work is the Pacific Coast Borax Plant, Bayonne, New Jersey because this building withstood a tremendous fire in 1902 (Gordon and Malone, 1994). According to Bradley, concrete of many types had been around since the Hellenistic –period (323-30 BC (Starr, 1991)). Concrete is described as being "a mixture of cement, water, sand, and sometimes an aggregate of crushed stone or gravel." This mixture, while not yet solidified, was taken and poured into molds to set up in the wanted form (Gordon and Malone, 1994).

Bradley (1999), notes "Reinforced concrete has provided for the manufacturer an entirely new building material. Indestructible, economical, and fireproof, it offers under most conditions features of advantage over every other type of construction." Bradley (1999), writes:

8

"Reinforced concrete was adopted for industrial buildings in the United States at the beginning of the 20<sup>th</sup> century, even though a few experimental buildings had been erected during the 1890's. An important impetus for the development of the new material was the improvement of processing methods for Portland cement that took place during the 1880's and 1890's. By 1900, engineers were discussing the advantages of reinforced construction; it didn't take them and the cement manufacturers long to convince industrialists to try the new material. By 1905 reinforced concrete construction had moved out of the experimental stage. In that year the New York City Department of Buildings approved the use of the Ransome system of reinforced concrete construction."

#### Prominent Historical Architecture

The oldest pyramids in Egypt were under construction around 2,700 B.C. The traditional style of pyramid, like the pyramids in Giza, was under construction for approximately a thousand years, until about 1,700 B.C. These pyramids adhered to a general composition where the inner parts were made of local limestone and the outer surface was made from a higher-grade limestone to give a "white sheen." The capstone was ordinarily cut from a harder grade stone like basalt or granite so that it could be plated with gold, silver, or electrum (McCauley, 2008). The oldest use for lime dates back to around 4,000 B.C. when the Egyptians used it to coat the outer surfaces of their pyramids. However, it is not easy to say when limestone was first used in mortar because written records were few until Vitruvius (Rowland and Howe, 1999).

According to professor Harley J. McKee (1961) the Erie Canal was built in order to connect the Great Lakes to the Hudson River. This canal extended more than 350 miles and used seventy-two locks. McKee (1961) describes the canal as: "Its general section maintained a depth of water of four feet, twenty-eight feet wide at the bottom and forty feet at the surface; the banks were of earth. Locks were twelve feet clear in width and ninety feet long. Those of the middle

section of the canal had stone walls six feet thick, on a foundation of hewn timber one foot thick, over which were laid well-jointed three-inch planks. The canal passed over a number of streams by means of wooden aqueducts supported by stone piers. Natural cement was employed in the mortar of these and other stone works, particularly the portions under water (McKee, 1961)." This major undertaking was made possible through surveys made in 1808 by James Geddes. Eight years later in 1816 the New York State Legislature passed a bill allowing the preparations of plans and cost estimates. The project was broken up into three pieces. The middle ran from Rome, NY to the Seneca River. Benjamin Wright, chief engineer, and his assistant Canvas White were in charge of constructing this section. Wright's section officially began on July 4, 1817 (McKee, 1961). McKee (1961) states that Wright wanted to import limestone; specifically Tarras or Roman cement, of the appropriate quality for the construction of the Erie Canal but this expense was not allotted in the budget (McKee, 1961). Therefore it was imperative that a local limestone of equal quality was found to ensure that faulty construction did not persist, as was the case in 1792 with the Inland Canal. According to McKee, in the town of Sullivan one such limestone was found and used in the canal construction. From a report dated January 25, 1819, the following was written: "...we export to make a very important use; as, by a number of small experiments, in which, after being thoroughly burnt and slaked, or ground, and mixed in equal portions with sand, it appears to form a cement that uniformly hardens under water..."(McKee, 1961).

#### Historical Milestones of Mortar Development

The use of natural cements date back to the Roman Empire. Vitruvius, a major architect during the period who wrote ten books on architecture, describes the necessary building materials needed to produce quality concrete for building in a variety of situations. He goes into great detail when describing the type of sand and lime, as well as the process for burning limestone. In a translation of his original book, Rowland and Howe (1999) state, "In concrete structures one

10

must first inquire into the sand, so that it will be suitable for mixing in with it." "These are the types of excavated sand: black, white, light red, and dark red. Of these the type that crackles when a few grains are rubbed together in the hand will be the best, for earthy sand will not be rough enough" (Rowland and Howe, 1999). Vitruvius even indicates where to look for quality materials when there are no visible, surface sand available. "If there is no sand beds where it may be dug out, then it will have to be sifted from riverbeds or gravel deposits, or, of course extracted from the seashore (Rowland and Howe, 1999)." However, Vitruvius does specify which sands are better. He makes it clear that "excavated sands" are better than "extracted" sands. Vitruvius states, "Excavated sands, on the other hand, dry quickly in construction, and the plastering stays in place; they will also bear ceilings, but only those sands that are from newly discovered sand deposits" (Rowland and Howe, 1999).

Vitruvius also references which stone is the best for making lime. "...then we must be careful about our lime, and whether it has been cooked down from limestone or silex (hard limestone). And that which is made from denser and harder stone will be useful in construction, and that made from porous stone, for plaster. When it has been slaked, then the materials should be mixed so that if we are using excavated sand, three parts of sand and one of lime should be poured together. If on the other hand, it is river or sea sand, two parts of sand should be thrown in with one of lime. In this way the rate of mixture will be properly calibrated. Furthermore, if one is using river or sea sand, the potsherds, pounded and sifted, and added to the mixture as a third part, will make the composition of the mortar better to use (Rowland and Howe, 1999)." While describing which stones are best for creating lime and in what ratios they should be mixed, Vitruvius describes the kilning process and it's importance. "If on the other hand, we throw it (stone) into the kiln, then, caught up in the flame's intensity, it will shed its original property of hardness, and with its strength burned away and sucked dry, it will be left with wide-open pores and voids. Therefore, with it air and water burned away and carried off, it is left with a residue of

latent heat. When the stone is then plunged in water, before the water absorbs the power of its heat, whatever liquid penetrates into the pores of the stone boils up, and thus by the time it has cooled it rejects the heat given off by lime. Therefore, whatever the weight of stones when they are cast into the furnace, they cannot have retained it by the time they are removed; when they are weighed, although their size remains the same, they will be found to have lost a third part of their weight because of the moisture that has been cooked out of them. And thus, because their pores and spaces lie so wide open, they absorb the mixture of sand into themselves and hold together; as they dry, they join together with the rubble and produce the solidity of masonry" (Rowland and Howe, 1999).

Canvass White began his career as an assistant engineer for Benjamin Wright. Wright was in charge of the construction of the Erie Canal when the project started in 1817. Building the canal with the appropriate materials posed a problem. The project was in need of economic hydraulic cement. Loammi Baldwin had volcanic ash shipped in from the West Indies because the cost to import from Europe was costly. As the project continued, more cost effective cement grew in importance (Howe, 2007). Wright encouraged the discovery and selection of native limestone fit for use as hydraulic cement. Canvass White, aided by his experience in England, was capable of identifying the acceptable limestone required to make cement with the required specifications. White was also encouraged by Governor Clinton to review and uncover as much as possible about canal construction. White traveled to England and reviewed some 2,000 miles of canal, talking with engineers and making accurate drawings. White also bought state of the art surveying equipment and asked questions of the builders and engineers about their limestone and mortar (Mckee, 1961). White found a European-type limestone in Chittenango, New York and was awarded his patent in 1820 for three operations: quarrying, burning, and grinding of the limestone. The limestone was quarried by hand, the rock was burned in kilns fueled by wood, and was ground again using "water-powered trip hammers and hand hammering (Howe, 2007)."

According to Howe (2007), gristmills were also used to reduce the burnt limestone into a powdered form.

Approximately thirty years prior to White, James Parker patented his "Roman Cement" in 1791. However, it was not until 1796 that Parker received another patent for "A certain Cement or Terras to be Used in Aquatic and other Buildings, and Stucco Work (Elliot, 1992)." According to Elliott (1992), Parker produced his cement from local areas along England's coast. Particularly in areas that had "low cliffs where London clay reaches the shore." These deposits came where harvested by hand as they were "kidney shaped stones." These stones were then smashed by hand to harvest the clay-like material from veinous deposits. The next step was placing the material in bottle-shaped kilns after they had been lit for three days and were fueled by coal. The material was then ground with millstone, sieved, and then placed in barrels for shipping (Elliot, 1992).

During the same decade that White was producing his cement in New York, Joseph Aspdin, a bricklayer, patented his own cement that he called "Portland cement (Elliot, 1992)" in 1824. According to Elliot (1992), Aspdin gave his cement this name because it was said to have the same strength and look as that found in the town of Portland located on the southern coast of England. Elliot (1992) goes on to say that the modern definition of Portland can be interchanged with Aspdin's cement for the simple fact that the technology did not exist that would allow the lime to be cooked at the high, steady temperatures that the modern Portland cement requires. Elliott (1992), goes on to describe Aspdin's kilns as being bottle-shaped, fueled by coke, and being approximately thirty-six feet in height and seventeen feet wide. Elliott (1992) adds that, this burning process caused the lime to cook unevenly because the entire brick kiln had to re-heat and thus caused a costly hand sorting and inspection.

13

#### Natural Cement Classification and Process

In Europe, natural cements were the choice of builders and architects and appeared as early as 1796 (James Parker's patented Roman Cement). Natural cements were used at great length during the 19<sup>th</sup> century. In 1824 Portland cement surpassed natural cements with Joseph Aspdin's ordinary Portland cement. Portland cement soon over took natural cements because new technologies allowed it to be made stronger and water proof (Varas, 2005).

According to Varas (2005), natural cements were composed of a marl comprised of 75%-60% carbonate, 25%-40% clay, and were cooked at a temperature between 800 and 1200 degrees Celsius for 8-12 hours. Natural cements provided an advantage over hydraulic limes because the hydration process takes place simultaneously and is takes less time to set, usually less than twelve hours. According to Ries and Watson (1936), hydraulic cements have "an increase in clayey and siliceous impurities, the burned rock show a decrease in its slaking qualities and develops hydraulic properties, or sets when ground and mixed with water. This product is the hydraulic cement, whose setting properties are due to the formation of new compounds formed during manufacture or when mixed with water. The new compounds formed in burning are probably solid solutions of aluminates and silicates of lime. Hydraulic cements can be divided into the following classes: hydraulic limes, natural cements, Portland cements, Puzzolan cements and high alumina cement. These five classes differ in regard to the new materials used, method of manufacture and properties of the finished product." Natural cements are categorized as being, "...made from a clayey limestone containing 15 to 40 percent of clayey impurities...(Ries and Watson, 1936), additionally, "Natural cements then are made from natural rock...They set rapidly and do not develop as high a tensile strength as the Portlands (Ries and Watson, 1936)." In the 19th century natural cements were used in Spain to construct ports, channels, drains, and water supply networks. Varas (2005), classifies natural cements into two categories: Rapid Natural Cements (RNC) and Slow Natural Cements (SNC). A RNC is characterized as having low clay

14

content (25-30%) with a cooking temperature between 1000-1200 degrees Celsius for a time between 12 to 20 hours. Natural cements characterized as an SNC have higher clay content of 40%, were cooked for 8-12 hours at temperatures between 800 and 1000 degrees Celsius.

#### Scientific Techniques and Testing

#### Modern Testing

Scientific testing goes hand-in-hand with the study of historic and ancient mortars. For example, Stewart and Moore (1982), tested eight mortar standards used to test three chemical techniques. The study used three techniques from H. Jedrzejewska, 1960, E.B. Cliver, 1974, and The American Society for Testing and Materials, Designation C85-66, 1971, (Stewart and Moore, 1982).

The Jedrzejewska (1960) method classifies large numbers of samples to help date the samples in comparative studies. This method determines: "by volumetric analysis the carbon dioxide content which is mathematically converted to calcium carbonate content, by gravity the sand content, and finally by difference the content of complex silicates (soluble fraction)." Stewart and Moore (1982), state, that "a high amount of complex silicates indicates a high hydraulic component and that a low level leads to low amounts of hydraulic components (Stewart and Moore, (1982))."

The Cliver (1974) method differs from Jedrzehewska's (1960) and The American Society for Testing and Materials because he uses gravity and color of fine residues to determine three numerical values. These values are: "the soluble fraction (lime and Portland cement soluble), the sand fraction, and the fine residue fraction." Stewart and Moore (1982), state "Cliver (1974) assumed that 40% of Portland cement placed in acid is insoluble and thus knowing the amount of fine residue present the amount of Portland cement in the sample can be calculated." The authors go on to say that if the fine amounts range in color from red to light tan then it comes from a clay mixture. Similarly, "if the fine amounts are brown from levels approximating the lime content then it came from cement. This method measures: percent lime, percent sand, and one of: percent Portland, percent natural cement, or percent clay (Stewart and Moore, (1982)."

The American Society for Testing and Materials (1971) uses gravity to measure the amounts of soluble silica present in samples. It is assumed that Portland cement contains 21% soluble silica and thus can be calculated. This differs from Cliver's (1974) method because Cliver (1974) assumed that 40% Portland cement in acid is insoluble. This study used laboratory prepared samples to duplicate the 19<sup>th</sup> and 20<sup>th</sup> century historic mortars found in Canada. The following criteria were met: the material was homogeneous, the nature and amounts must be accurately known, and standards have not undergone leaching or contamination.

The results of Stewart and Moore's (1982) indicates, that of the three tests, H. Jedrzejewska (1960), was the only one that was applicable because "of the characteristics that it measures, it does so accurately." The method of Cliver (1974), did not work because it wrongfully identified some standards as having Portland cement and one sample of having lime mortar with a clay residue instead of a hydraulic mortar. According to Stewart and Moore (1982), the American Society for Testing and Materials correctly identified the samples that are were designed to identify samples containing high silica content other than Portland (Stewart and Moore, 1982).

#### Thin Section Testing

In order to determine size, ration, and origins of limestone and mortar samples, the study of cement thin section samples may be used. "Thin section petrography is polarized light microscopy of rocks and other mineral-containing materials, using samples ground to the standard thickness of 30 microns (um) (Reedy, 1994)." Reedy (1994), states, "archeologists, and conservationists use this practice to describe and classify rocks, soils, and sands…and to study

many inorganic materials used in the production of cultural object," respectively. For example, archeologists use this method when studying clay cores of bronze statues, when looking for quartz content. Quartz makes up the largest component of clays or sediments and as such is used as a source indicator in geology. Studying the quartz through thin section petrography allows the determination of: size, shape, and texture. Determining these factors has been shown to aide in provenance work (Reedy, 1994). Similarly, Reedy (1994), also uses this technique on plasters and cements. For example, plasters are comprised mostly of burnt lime, gypsum, clay, sand, water, and organic materials. This process is used to identify the various materials that make up a plaster and their respective ratios. Reedy (1994), determined the size and impurities that may be present in the sand or lime. Reedy (1994), and Goren and Goldberg, (1991), indicate that thinsection petrography can be more useful than other techniques, like x-ray diffraction and scanning electron microscopy, because the thin-section analysis can show the grain textures and interrelationships between components. Similarly, a study by Hyman (1997), indicates that thinsection analysis was more useful in characterizing calcareous cements that were used in Hispanic Mesoamerican buildings. This analysis was able to identify microfossils, grain size, and the degree of rounding (Reedy, 1994).

#### Limestone Classification

The carbonate minerals in the limestone available to build these forts fall into two main classifications: magnesium calcite and pure calcite. Calcite contains a higher concentration of carbonate and this makes it more suitable to use in producing mortar (Ries and Watson, 1936). Since both dolomite and calcite are classified as limestone, both can be used effectively to produce mortar or as a building stone. However, dolomite is slightly higher on the hardness scale 3.5-4.0 versus calcite at 3.0. Ries and Watson point out that, "Both limestones and dolomites of dense and massive character, as well as those free from mineral impurities, are of good durability, although not as long-lived as dense sandstones or granites. Limestones weather primarily by

17

solution, that is to say, rain or surface water may slowly attack the rock, but the solution of the surface is likely to go on very unevenly (Ries and Watson, 1936)." Ries and Watson (1936) make the determination that dolomite is a slightly better choice by stating, "Dolomites do not weather so readily by solution." The following shows the difference in composition between these two forms of limestone:

#### **Calcite**

This is the mineral form of calcium carbonate (CaCO3). This mineral has the proportions of CaO-56% and CO2-44% (Ries and Watson, 1936). Ries and Watson (1936), state, "Rocks composed chiefly or entirely of calcite have varied uses, principal among which may be mentioned the manufacture of natural and Portland cement, the manufacture of lime for mortars and cements, and for agricultural purposes, as a fluxing material in blast furnaces, as ornamental and building stone, etc".

#### **Dolomite**

According to Ries and Watson (1936), dolomite is, "A carbonate of calcium and magnesium, CaMg (CO3) 2. Carbon dioxide 47.9, lime 30.4, magnesia 21.7." Ries and Watson go on to describe dolomite as: "A dolomite is similar in color, texture, and other physical characters to limestone, except that it is slightly harder, somewhat more resistant, because it is less soluble, and does not effervesce except feebly in cold acid. It is not always an original rock, but has sometimes been derived from straight calcic limestones by the substitution of magnesium carbonate for a part of the calcium carbonate-a process known as dolomitization. It is also used for flux and lime making. (Ries and Watson, 1936)."

#### **X-Ray Diffraction**

X-ray diffraction is used in tandem with thin section analysis to test the qualities of mortar and limestone at the composition of cements. Mortar analysis often uses several different techniques to determine different unknown characteristics of a sample. The common methods are petrographic analysis, x-ray diffraction, and acid digestion. The most useful method has been shown to be petrographic analysis (thin sections (Schnabel, 2009). However, the second most used is that of x-ray diffraction (Schnabel, 2009). X-ray diffraction (XRD) takes the mortar sample in powder form and identifies the crystal structures found in the sample. According to Schnabel (2009), this is considered to be a qualitative technique but with the use of certain algorithms has been shown to produce the composition of mortar binders. Schnabel (2009), points out that XRD does have difficulty with identifying different minerals with similar crystalline structures or arrangements. This same technique was done on stucco samples from the College of Charlestown and according to Krotzer and Walsh (2009); the XRD tests are able to produce precise, clear quality of mortar components. However, there is no technique that can produce analytical evidence of mortar components and proportions. Interpretation can come only from an experienced materials scientist (Krotzer and Walsh, 2009). This technique sends x-rays through a randomly oriented, powdered sample and shows the "regular spacing of atoms as the rays are diffracted by the crystal structure of the component mineral (Krotzer and Walsh, 2009)." XRD is very capable of determining the major, abundant minerals before the overlapping in wavelengths becomes a problem. Krotzer and Walsh (2009), state, "XRD is best suited to answer specific questions about mineral phases in portions of mortar that have been isolated by careful subsampling. XRD is not a tool to diagnose an entire mortar sample in order to generate precise information about overall composition or quantities of ingredients (Krotzer and Walsh, 2009)."

#### CHAPTER III

#### MATERIALS AND METHODS

#### Sampling Procedure

In order to determine the consistency of the mortars for each site, several tests and methods were conducted. First, initial samples were obtained from each site (Figures 3-19). Samples were taken from the interior walls from the oldest known structures. Samples were taken from interior walls and windowsills. Also, samples of limestone and sand were taken from each site to compare to the quality of limestone and sand found in the thin sections. Of the numerous samples (54) taken for all sites, eighteen were selected based on their potential for being the most representative and oldest at each site. These samples range in size from quarter (2.5 cm) to half dollar (3.1 cm) in diameter. Thin section mortar analysis was conducted on these chosen samples. These samples came from a variety of structures and positions. The eighteen samples, labeled PDH 1-18, came from Forts Washita, Towson, and Gibson. The following is the sample identification, listed in Table 1.

Sample I.D.	Location	Description
PDH 1	Washita	North Wall corner of Colbert House ruin
PDH 2	Washita	Interior south wall adjacent to east wall of Colbert House
PDH 3	Washita	Site 2 of same wall
PDH 4	Washita	Interior south wall of Colbert House
PDH 5	Washita	West window of Colbert House under window sill rock
PDH 6	Washita	Chimney interior east side
PDH 7	Washita	Hospital south east corner
PDH 8	Washita	Hospital back-middle outside wall
PDH 9	Towson	Kiln site 1
PDH 10	Towson	Kiln site 2
PDH 11	Towson	Kiln site 3
PDH 12	Towson	Adj. officer's west wall of garden
PDH 13	Towson	Adj. officer's west wall of garden
PDH 14	Towson	North west barracks
PDH 15	Towson	South west barracks
PDH 16	Gibson	Bement's 2008 mortar from brick #6
PDH 17	Gibson	N11 E99 (5-28-08) mortar
PDH 18	Gibson	N4 E20 20-30 cm (5-29-08) mortar

Table 1. Sample identification, fort, and description of mortar sample used in this study.

#### Fort Location at Geologic Setting

### Fort Gibson

Fort Gibson (Figure 2) is located on the flanks of the Ozark Uplift on rocks of Pennsylvanian Atokan and Morrowan Series (Huffman, 1958). The Atokan is sandstone, shale, and thin silty limestones. The Morrowan rocks include the Bloyd Formation, which is blue-gray dense limestone interbedded with dark gray shale (Huffman, 1958). Material for mortar production is mostly of poor quality at or near the grounds of the fort. The most suitable material was found approximately four miles up stream, along the bank of the Grand River. This material was harvested, placed in keels, and moved back down stream (Quartermaster General Consolidated files of Correspondence, 1832). Figures 3 and 4 show the footer of the original wall that was excavated June 2010.

#### Fort Washita and Towson

Forts Washita and Towson, both have similar limestone sources. Both forts are located on the former coastal plain and sit upon Cretaceous limestone and shale. This limestone is very high in CaCO3 and is thus very suitable for harvesting material in order to make quality mortar. This limestone is readily available in the immediate vicinity of these forts (Puckette, 2010; Huffman, 1958). Figures 5-9 show exterior wall and structures that were used to gather samples for testing at Fort Washita. Fort Towson samples locations are noted in Figures 10-14. Figures 10 and 11 show the original, freestanding kiln site at Fort Towson. Similarly, Figures 12-14 are barracks ruins from Towson that were also used in the sample process.

The following map illustrates the generalized geology of eastern Oklahoma and the locations of these three forts (Puckette, 2010), (USGS, 1960):

#### Eastern Oklahoma

Gravel and sand (Recent) Limestone, shale and sandstone (Cretaceous) Shale, sandstone and limestone (Upper Pennsylvanian) Shale, sandstone and limestone (Middle Pennsylvanian) Shale and sandstone with coal and limestone (Middle Pennsylvanian) Shale and sandstone with coal and limestone (Lower Pennsylvanian) Limestone and chert [Ozark Uplift] Mississippian



N

Kay

Noble

(Ottawa)

Delaware

Adair

Craig

Mayes

Nowata

Rogers

Wagoner'

Muskogee

Washington

Tulsa

Osage

Creek

Pawnee

Payne

Lincoln

(After USGS, 1960)

Figure 2. Generalize geologic map of eastern Oklahoma showing the locations of forts examined in this study.



Figure 3. Original footer for wall at Fort Gibson that extends past current boundary of reconstruction.



Figure 4. Excavations during 2008 Oklahoma Archeological Survey field season at Fort Gibson.



Figure 5. Original barracks, later used by the Colbert family at Fort Washita. This building was twice destroyed by fire.



Figure 6. Interior of barracks showing stone work of the 1st reconstruction at Fort Washita.



Figure 7. Stone wall constructed of the local limestone. Mortar samples were obtained from the original wall and were1st generation mortar at Fort Washita.



Figure 8. multi-generational mortars in stone wall at Fort Washita. Samples were restricted to 1st generation mortar behind later pointing.



Figure 9. Foundation of Fort Washita post hospital.



Figure 10. Top view of lime kiln at Fort Towson. This structure is mostly original, but contains some re-pointing.



Figure 11. Interior of lime kiln at Fort Towson.



Figure 12. Original wall of officer's quarters at Fort Towson.



Figure 13. Partially re-constructed barracks wall without pointing at Fort Towson.



Figure 14. Fragment of 1st generation mortar from barracks chimney at Fort Towson.

#### Thin-Section Analysis

#### Description of General Thin-section Process

Using thin section analysis to determine original mortar composition of historic structures is a common methodology. Mortar samples are taken from prospective sites and produced into thin sections (30 microns thick) and mounted to a slide and analyzed through a polarizing microscope (Scholle, 1979). Then samples are initially viewed for distinct characteristics such as grain size, roundness, color (with and without polarized light), and pore space or voids. Thin section analysis also allows for a more tedious and rudimentary skill in determining composition. A point count is conducted on each slide; putting each slide into a coordinate plane and along the determined axis the mortar components are counted including grain size, shape, roundness, color, and pore space.

#### Photographing Thin-Sections

Thin-sections are the slides that were made of rock and mortar samples from each of the three sample sites: Fort Gibson, Towson, and Washita. Mortar samples were taken from the oldest known structures; also natural sand and limestone samples were taken. Of the fifty samples taken from all three sites, eighteen were selected for their age and representative population. These samples that were selected came from various buildings and differing locations, such as an interior wall or foundation.

Each slide (thin section) was placed under a polarizing microscope with a digital camera. The 2X microscope objective was chosen, and photographs were taken of five sample areas, determined at random, for each slide. This results in a total of eighty-five sample areas, with the exclusion of slide 12, which was omitted due to its damage. Each sample area photograph was laser copied onto an 8.5x11 inch piece of paper to make it easier to count grains, void space (porosity), and mortar. A ¼ inch grid was created and transferred onto a transparency and placed over each sample area. Each sample area was divided into quadrants: Top Right Quadrant (TRQ), Bottom Right Quadrant (BRQ), Bottom Left Quadrant (BLQ), and Top Left Quadrant (TLQ). Each quadrant was 130 units (10x13) in total area. Each photograph (sample area), once laser copied measured 6 ¾ inches in length and 5 1/64 inches in width. The sample areas, slides 1-18, are counted and totaled. These figures are then tabulated for the whole slide and compared to the other seventeen slides. These photographs (sample areas) were separated by fort location. For example, slides 1-8 originated from Fort Washita, 9-15, excluding 12, were retrieved from Fort Towson, and 16-18 came from Fort Gibson. Thus, comparison between sample areas in each particular fort and comparison between the actual forts became possible

Each slide had five, random sample areas photographed for a total of eighty five total sample areas. Each sample area, when photographed, with a 1mm scale bar burned into the image on the bottom right quadrant. A total of 44,200 grid squares were counted to determine percent grain, mortar, and void space.

#### Count Parameters

Similarly, each thin section was placed back under the polarizing microscope and recounted. This count was preformed to gather 100 random grain samples and classify them by size (mm), angularity, and identification (microcline feldspar, plagioclase feldspar, quartz etc.). The microscope was used with the 10-power lens, 8x eyepiece, and each slide was counted. The total number of counted grains was 1700 as slide #12 was irreparably damaged. The following are example images slides typical of those that were counted:



Figure 15. Side-by-side examples of the differences between two samples from the same location. Images were taken using plane-polarized light (PPL).

#### Calcium Carbonate Equivalent Measurements

#### Modified Pressure Calcimeter Test

The mortar samples were also subjected to an experiment to determine their inorganic calcium carbonate equivalent (CCE) content. This test was conducted using the modified pressure calcimeter method. This method uses volumetric displacement to calculate inorganic calcium carbonate. This test is developed from the reaction from HCl and carbonates and it measure the loss of CO2. It also, uses the equations developed from Loeppert, Suarez, and Wagner. The results range fro .25- 100% CaCO3 for a 20 mL serum bottle, which is used as the reaction site. For 100 mL bottle, which are the bottles that were used for this project, result in ranges of 2.00-100%. The equipment used was: analytical balance: 100g capacity, repipette dispenser, calibrated to 2.0 mL, 100 mL wheaton serum bottles, .5 dram vials (2.0 mL capacity), gray butyl rubber stoppers, tear off aluminum serum bottle seals, hand crimpers, power supply (24 volt DC. -2amp.), digital voltage meter, capable of reading .01 volts resolution, and a pressure transducer 0-105 kPa (Setra Model 280E).

The reagents used in this method are: deionized water, ASTM Type I grade, Calcium Carbonate (CaCO3) fine ground (100 mesh sieve, 150 um), reagent grade, Hydrochloric acid (HCl) 6N with 3% by weight ferrous chloride). 500 mL of HCl was transferred to 400 mL of deionized water and adds 30g of FeCl2\* 4H2O and brought to 1.0 L volume with deionized water. Due to the fact that the percentage of CaCO3 was higher than 30%, 100 mL serum bottles were used with standard concentration percentages of 10, 20, 30, 40, 50, and 80%.

The procedure for this method was conducted according to the following:

 Weigh 100g of soil into a 100 mL Wheaton serum bottle. Place CaCO3 appropriate standards in 100 mL Wheaton serum bottle.

- Pipet 2.0 mL of 6.0N HCl reagent into .50-dram glass vial. Gently insert acid dram vial into reaction vessel with sample, but do not allow solution to mix with the sample. Cap reaction vessel with gray butyl rubber stopper and crimp with aluminum tear-off seals.
- 3. Shake reaction vessel vigorously to ensure that acid solution in the dram has mixed with the soil. Run three blank (1.00g of laboratory sand with acid vial) with each analysis run.
- Prior to reading samples with pressure transducer (15 minutes), rotate the acid along the sides of the reaction vessel to ensure that soil on the sides is reacting with the acid.
- 5. After two hours of reaction time with the samples and standards are ready to read on the pressure transducer.
- Record the voltage output to two decimal places. Subtract the average voltage of the blanks from the standards and samples to obtain the change in pressure due to CO2.

The calculation for this method uses linear regression to determine the slope (regression coefficient) and the intercept (b) of the curve of pressure change versus the dependent variable of percent CaCO3. Inorganic carbon can be obtained by dividing the formula weight of CaCO3 (100) by the formula weight of carbon (12) and multiply this by the percent CaCO3. The following are the equations used to calculate total inorganic carbon:

% CaCO3= (regression coefficient)\*(delta pressure in volts) + b

% Inorganic carbon= % CaCO3/ 8.33

#### Inorganic carbon $g/kg = 10^*$ (Inorganic Carbon %)

The aforementioned scope, reagents, procedures, and calculations were recorded from Loeppert and Suarez, 1996.

#### Calcium Carbonate Equivalents by Titration

Samples of limestone were also taken in order to determine their Calcium Carbonate Equivalent (CCE) to determine their level of purity. This was done because it has been determined that limestone with a purity level of 60-75% was preferred for mortar production. Seven samples were prepared and sent to The Oklahoma Department Agriculture's Food and Fertilizer Lab for CCE analysis (Loeppert and Suarez, 1996). These seven rock samples, all from fort sites, were smashed into gravel size elements. These gravel size elements were then resmashed with a three-pound mallet to put them into manageable size elements to be placed in a ball mill. Once small enough, samples were placed into a ball mill and turned into a fine powder. Those powdered samples were then mailed.

#### Historical Documentation

Historical documentation from the National Archives in Washington D.C. was retrieved in an attempt to find an historical link to where and when the construction materials for these sites were acquired. Also, due to the fact that Fort Towson is the only site with a visible, intact kiln, documentation was acquired to possibly locate any kiln sites for Forts Washita and Gibson. Fort Washita does have a rubble pile that is said to be the remnants of the kiln, but historical documentation is needed in order to do away with conjecture. Old post records were pulled in order to determine day-to-day activities to piece together a time frame from post construction to completion, when the primary and secondary structures were built and what those were. Records were also pulled to find how many of these resources were shipped in versus what resources were locally obtained. For example, was the lime used for the mortar harvested from local limestone and produced on site or was it shipped to the posts via river and tributary systems? The same records should also show how many or what time of year these shipments took place. Similarly, personal correspondence was obtained to get an accurate portrayal of how much communication went on between these frontier posts (post-post) and Washington D.C. This may be important to determine how much leeway the post commanders had in the construction of their posts, which should determine how much of the resources were shipped to their locations with in time and budget constraints.

#### CHAPTER IV

#### **RESULTS AND DISCUSSION**

#### Source Limestones for Fort Mortar

#### Calcium Carbonate Equivalents

The Fort Gibson sample, SL 1, contains more than the minimal CCE required to produce quality mortar on the frontier. The other two Fort Gibson samples, SL 2 and SL 3, failed to reach the minimum requirement of 60% CCE for mortar quality (Table 2). Samples SL2 and SL3 could have been used for temporary structures that were used and manned while permanent fortifications and structures were being built. The limestone samples from Fort Washita, SL 4 and SL5, both measured sufficiently higher than the required percent of CCE for mortar. Table 2 shows the Fort Towson samples, (SL6 and SL7), were also sufficiently high in CCE to be used as mortar. Table 2 demonstrates that Forts Washita and Towson both yielded high CCE percentage is congruent with pure limestone sources from the nearby Cretaceous deposits that the two forts are located. The CCE levels for the source limestone were done in hopes that a comparison between the forts could be made. There are some pre-existing circumstances that prevented this analysis from taking place. Fort Gibson is the fort that has the most attention. It benefitted from a 1930's WPA project that built a replica of the original fort. Also, most if not all, of the

structures currently standing are the results of decades of modern repairs. For example, the stone used for the current structures was moved from various areas all over the grounds and modern cement was used to adhere them. There is too much human traffic to get a reliable result from the CCE readings. Fort Washita has a similar problem to Fort Gibson. After the military abandoned the grounds, it became a private family home until the latter 20<sup>th</sup> century. The main barracks that was sampled showed clear evidence of at least three generations of mortar being used. It was this barracks that was turned into the main family home, so too much contamination of the grounds made this comparison difficult. Fort Towson is the only place that has remained un-touched from recent contamination. The For Towson site has a freestanding kiln that is in its original state. The barracks, officer housing, and other structures have been left in their original state with very little attempt of reconstruction. This fort provided the best environment for comparison. It is for these reasons that the CCE test does not adequately answer which fort was constructed better and with what materials.

Sample #	Location	<u>CCE %</u>
SL1	Fort Gibson	93.91
SL2	Fort Gibson	40.52
SL3	Fort Gibson	28.75
SL4	Fort Washita	109.63
SL5	Fort Washita	112.66
SL6	Fort Towson	112.7
SL7	Fort Towson	110.04

Table 2. Calcium carbonate equivalents (CCE) for limestone samples from Forts Gibson, Washita, and Towson.

#### X-ray Diffraction Analyses

The results are slightly different for each fort, but all three forts tested for the presence of calcite. This is congruent with the calcium carbonate equivalents that were tested (Table 2). As demonstrated in Figures 16-18, all three sites contain calcite, a specific limestone mineral made primarily of 56% CaO and 44% CO2. This coincides with prior CCE tests that show Fort Gibson samples testing less than the required minimum of 60% CCE. Calcite must measure 100%; if the readings are greater than 100% (i.e. SL4-7) then the sample contains dolomite. Dolomite is a mineral containing magnesium that often occurs in limestone. However, Forts Towson and Gibson show no peaks for pure calcite, only dolomite (calcite magnesium). Regarding mortar production, dolomitic limestone would have been preferred over calcitic limestone. Dolomite is harder than calcite and thus would survive the elements and environment better. The analysis of samples from Fort Washita did not contain any indication of dolomite. This is most likely the result of the absence of dolomite in the limestone used during construction.



Figure 16. x-ray diffractogram for limestone for limestone sample #SL1 from Fort Gibson.



Figure 17. x-ray diffractogram for a limestone sample #SL5 from Fort Washita.



Figure 18. x-ray diffractogram for a limestone sample #SL7 from Fort Towson

#### Thin Section Analyses of Mortar

#### Grain Counts-Fabric Analysis

Fort Gibson's microscope point counts of mortar, grain, and voids for thin sections are significantly smaller than Forts Washita and Towson because there were fewer samples to utilize for the fabric analysis (Table 3). Fort Gibson did not have the amount of mortar to sample that Forts Washita and Towson offered. Fort Gibson was the location for a WPA project in the 1930's and as such was too contaminated from the re-building process. Stones and other materials were moved from one fort to re-build other post structures. Due to the fact that each fort has a minimum of three variables (grain, mortar, and void space), it is easy to see how closely related each fort is. The samples from each site vary; each fort's mortar was virtually produced with the same method.

Location	Point Count of Content			
	<u>Grain (%)</u>	Mortar (%)	<u>Void (%)</u>	<u>TOTAL</u>
Fort Gibson	210 (41%)	273 (53%)	33 (6%)	516
Fort Washita	10940(53%)	8336 (40%)	1524 (7%)	20800
Fort Towson	7223 (46%)	7259 (47%)	<u>1118 (7%)</u>	<u>15600</u>
mean	6124 (50%)	5289 (43%)	892 (7%)	12305

Table 3. Fabric Analysis: Point counts of mortar thin sections from each fort.

#### Grain Counts-Identification and Size

The data in Table 4 shows that similar sized sand was used to make mortar at each fort. Each of the forts did have a variety of sand sizes. For example, close to 60% of Fort Towson's sand falls as medium sand, between .5 and .25 mm (Table 4). But the same data shows that both Fort Gibson and Washita have the closest sand size relationship; they are almost identical. Each fort had sand that was larger and smaller than the average, however, it is interesting that each fort averages in the medium sand size. This suggests that sieving may not have been used to attain a narrow particle size. This medium size that transcends each fort, must have been the known size requirement for mortar production. It is also possible that medium sized sands were as small as they could sieve with the known technology available on the frontier. Additionally, this count was carried out to identify any unique minerals that might prove as a geologic source marker (Table 5). The forts do share three rock and mineral: claystone, hematite, and dolomite. However, it is Fort Washita that shows a much more varied mineral and rock population. One explanation for Fort Washita's eclectic mineral and rock population is its geographic location. The Washita River that runs past Fort Washita is fed as runoff from the Wichita Mountains. The Wichita Mountains contain a variety of minerals and rocks primarily eroded from a granite source. This granite source gives Fort Washita its variation in mineralogy.

Location	mean size (mm)	<u>% Quartz</u>	% Other
Fort Gibson	0.35	90.67	9.33
Fort Washita	0.39	92.00	8.00
Fort Towson	<u>0.36</u>	<u>98.50</u>	<u>1.50</u>
mean	0.37	93.72	6.28

Table 4. Comparison between quartz and non-quartz elements and respective percentages.

Table 5. Percentage of major constituents in mortar as determined by point counts.

Rock/Mineral	Ft.Gibson%	Ft.Washita%	Ft.Towson%
claystone	3.70	0.25	1.00
hematite	0.67	0.17	0.88
dolomite	0.33	0.75	0.33
microcline	4.30	2.30	0
plagioclase	0.33	2.88	0
muscovite	0	0.13	0
siltstone	0	0.13	0
schist	0	0.13	0
hornblende	0	0.25	0
limestone	0	0.13	0
chalcedony	<u>0</u>	<u>0.25</u>	<u>0</u>
Total			
(% other)	9.33	7.37	2.21

Figure 18 shows that Fort Towson, for the majority, has medium size sands. This would suggest that this sand was pulled from a local source. Smaller sands (category 1 and 2) most likely came from local bodies of water. For example Fort Gibson stands on the Grand River, while Forts Washita and Towson are built along the Washita River and Gates Creek respectively. These sands would have a diameter less than 0.5 mm because they were more heavily eroded from traveling further downstream and ending up in smaller tributaries like Gates Creek. Forts Gibson and Washita both have coarse grain sand. This could suggest that this sand may have come from an outside source or strengthen the argument that sand was a commodity with which the builders were willing to purchase if it was of proper quality and standard. This goes with

documentation that discusses contracting sand for mortar at a price within the constraints of the military's budget





Figure 19. Sand size distribution for Forts Gibson, Washita, and Towson; 6=gravel (4-2mm), 5=very coarse sand (2-1mm), 4= coarse sand (1=0.5mm), 3= medium sand (0.5-0.25mm), 2= fine sand (0.25-.125mm), 1= very fine sand (.125-.0625mm).

#### Historical Documentation

#### Fort Washita

Virtually no documentation was discovered at the National Archives. I was able to find references to a Fort Washita. For example, it is obvious that a Fort Washita existed; however there many have been multiple forts with that name throughout the country. There may have been other forts with the same moniker. However, information in the way of personal correspondence or written records regarding construction was not found working with a constricted time element. Discovery of such information would be possible if one were able to spend three to six weeks going through the volumes of overt minutia, it is probable that information regarding the construction or how and when the materials were retrieved could be of importance.

#### Fort Gibson

Several correspondence were retrieved from the National Archives in Washington D.C. discussing where and how materials were to be furnished for construction. On 12, July 1832 Clark wrote to Jessup detailing where stone could be found for the construction of walls. He writes, "The stone necessary for the walls of this building can be obtained from a quarry about ½ miles distant and hauled in stone carts. These carts should be purchased at Pittsburg or Cincinnati as also the Iron Nail, Paints, Tools, & etc. and sent on with the subsistence stores early in March. The mechanics should also be employed at and of those places and sent in the (sand) boat as they can not be obtained in this country at any price." In the same letter Clark goes on to provide examples of how to ship lime for mortar and lumber goods as follows: "The lime can be made about four miles up Grand River on the opposite bank and transported in keels." The plank must come from the Mill below Fort Smith say Crawford Court House and Little Rock, by contracting this season they will have time to saw the plank and have it ready. The first rise of the water in the spring. This will be necessary or it cannot reach here the next season. The balance of lumber can be furnished by the labor of the troops within five miles. (Quartermaster General consolidated files of correspondence Misc M.F. No. 587 Roll 1).

Similarly on 30 June 1833, Clark writes Jessup again explaining where to find materials for construction. Clark states, "The Military reserve being mostly prairie very little timber suitable for building can be obtained. It must be purchased from the Cherokee Indians (on) the reserve must be enlarged. This can be done by purchase or lease." Clark continues, "There is no pine or other suitable timber for plank above Fort Smith. Consequently the Plank for building must be (hand) posted from below near 300 miles, at an expense of \$20 pr. feet. This with the

cost of the mill brings (this) to about \$40 pr. feet" (Quartermaster General consolidated files of correspondence Misc M.F. No. 587 Roll 1). He goes on to discuss prime location for finding building stone and lime. These are to be found four and seven miles up the river respectively and to be transported in the same fashion.

By 1845 the situation of finding construction materials had not changed that drastically. Collins writes to Stanton on 20 August 1845 that (Collins), "I have made contractors for lime and sand (after duly advertising for "proposals") the lime is to be delivered at 19 ½ cents per bushel, the sand at 5 ½ cents per bushel thus, you will see I have contracted for the delivery of these articles, at prices less than are put down in my estimates" (Quartermaster General consolidated files of correspondence Misc M.F. No. 587 Roll 2).

Some of these correspondences are relating to Fort Gibson as if it where in Arkansas. This makes sense because the original site for Fort Gibson was in Van Buren, Arkansas. However, the construction site was changed to where it sits currently. Also, the other correspondence relates to the current Fort Gibson site and this shows that methods for procuring materials for construction were very similar. This also makes sense because both sites where selected for their location to water and limestone.

There was no historical mention in any of the ones researched that mentioned a specific ratio or recipe to create the mortar used for Fort Gibson's construction. It is obvious that a kiln was needed to produce the mortar, however, no physical evidence has been found to date. It is hypothesized that a kiln was built further up river into a hill, where they would most likely be shipping sand or kilned limestone via barge.

#### Fort Towson

Quartermaster correspondence was obtained from a first lieutenant who wrote to Major General Jesup on 26 August 1835 explaining a list of contracted laborers who were to be employed "indefinitely." For example, an Irish immigrant named Elias Hughes was brought on to be the Clerk of the kiln and was to be paid a sum of twenty-five dollars a month. Similarly, people were brought in to be teamsters, a lawyer, a carpenter, and to be in charge of cattle (Consolidated Quartermaster Correspondence, Fort Towson, Box 1145, Entry 225.)

Similarly, correspondence from Fort Towson, in 1835, gives a detailed description of the dimensions and how the original structures were constructed. For example, the structures were erected in a square formation. This formation consisted of four block and the angles of these points to the companies, which was numbered at four. The hospital is south west of the square and has two rooms comprised of the wardroom and infirmary. The wardroom is forty feet by twenty-one feet with the windows facing forward, port windows in the rear, with a capacity of fourteen. The infirmary is thirteen feet by twenty feet (Consolidated Quartermaster, Fort Towson, Box 1145, Entry 225).

Approximately one year later, correspondence from Fort Towson states that several buildings: officer's quarters and kitchen would be in need of new roofs within the ensuing year. The barracks is said to be suitable for dimensions and comfort level, but one wall, which doubles as a "face of the fort," has fallen to the point that it will need to be repaired and is making the rest of the barracks unsuitable. However, on the wide scale, the repairs to the post remain at a minimal need (Consolidated Quartermaster, Fort Towson, Box 1145, Entry 225).

The historical record does not mention if outside sources were used to transport building material. However, this site is the only one with an original, freestanding kiln on its immediate grounds. This strongly supports the use of local, natural limestone to produce their mortar. The same may be inferred with the sand.

#### CHAPTER V

#### SUMMARY

Source limestone, from all three forts, was tested for CCE in order to determine if the forts possessed the minimum percent required for basic mortar construction. All of the forts had samples that met this 60% minimum. However, two samples from Gibson fell well under this required amount. This can be attributed to one root cause: heavy amount of recent, human involvement from the re-building of the original fort structures and the construction of the recreation of the original fort in the 1930s (Fowler, 2009).

X-ray diffraction tests were conducted on the samples created from the source limestone to determine their mineral composition and verify if the carbonate in the limestone was dolomitic or pure calcite. There is an engineering advantage to using dolomite over calcite because dolomite is harder and withstands the elements and natural erosions processes better than its softer counterpart. All three forts contained calcite but only two, Forts Towson and Gibson, indicated magnesium, but not stoichiometric dolomite. All three forts showed different phases of construction, but the oldest parts used the stronger, harder limestones in the earliest phase. One example of higher magnesium calcite limestones durability is the shape difference when both limestone types were cut into blocks. The magnesium calcite blocks held their right angles, while the pure calcite blocks had rounded edges. Thin section analyses, through the use of fabric identification and size analyses, were performed through microscope point counts. The fabric analysis yielded some very interesting results. Each fort showed tremendous similarity regarding the relationship between grain, mortar, and void space. Regardless of population size each fort had a mean relationship of 50% grain, 43% mortar, and 7% void space. Similarly, the identification and size analysis yielded a similar result. Each fort tested showed a sand size between .35 and .39 mm with an average of 93.72% identified as a quartz (sand) mineral. The later analysis proves that a sieving process had to have been used because sand would not have been produced naturally in that amount.

No definitive answer was found when procuring a formal recipe was not able to be determined for the period mortar from the National Archives. Each fort was built on a site that could provide water, quality limestone, and a plethora of sand. There are three hypotheses that were postulated at the beginning of this research. One hypothesis states that mortar-making materials were retrieved and used strictly from on site. The second hypothesis states all the materials were shipped in using the local river system. However, it is the third hypothesis that is the most acceptable. Local materials and out-sourced materials, as well as, local and out-sourced labor were utilized in the construction of these forts. This is shown to be true from several of the historical correspondence naming materials that were purchased for a price lower than the budget allowed. For example, at Fort Gibson lumber was contracted at \$20 (per thousand) foot from the Cherokee. Other materials such as limestone and building stone were brought from seven and four miles upstream from the Grand River. Additionally, sand and lime were being shipped to Fort Gibson as late as 1845. Both lime and sand were contracted out for 19.5 cents and 5.5 cents per bushel. Similarly at Fort Towson, certain job positions were filled from contract labor. For example, there is mention of hiring Elias Hughes as the Clerk of the Kiln at Fort Towson.

The main, underlying point of this research was to produce information so that interested parties could reproduce period mortar so that these structures would be preserved for future generations. The best way to accomplish this task would to recreate the needed equipment and materials. Some of these materials will differ for each fort but the equipment needed to prepare these materials will be uniform for each fort. The raw materials are limestone, sand, and water for slaking. Fort Gibson will be able to produce magnesium limestone and Forts Washita and Towson will be using a more calcitic limestone. These limestones are native to their respective areas and would have been used during the period. The same can be said for the sand. Each fort used local river sand to act as the binding agent with their mortars. A paramount piece of equipment is going to be a kiln. This process is what dehydrates the limestone, thus creating a pivotal point in the mortar process. I would recommend breaking or milling the limestone prior to cooking it in the kiln but that would be determined by how authentic a person wanted the mortar to look. For the period, they most likely broke limestone into manageable sizes to fit into the kiln and then broke further after it had cooked down. In order to cook the limestone, the kiln should be heated to around 1000 degrees Celsius and the rock cooked for eight to twelve hours. The limestone needs to be ground into a powder form, something that is easily mixed with water. The sand, from all three forts, consistently averaged 0.37 mm in size. This means a sieve will also be needed to ensure that the sand size is uniform. I would suggest using a number 40 or 45 sieve to get close for the appropriate grain size. Once the lime and sand are prepared mix the two in equal amounts and add water to consistency.

The aforementioned research contained in this thesis does not allow for a clear, concise answer. The requirements for determining an exact "recipe" will take many more samples and tests. However, this research data is a good starting point for the next researcher to begin and build onto these findings. If this material was to be enriched, it would be greatly beneficial to return to the National Archives and allow for a much more thorough investigation of the consolidated quartermaster rolls and the miles of microfilm left untouched for this purposes of this research. For example, I was only able to spend one week going through any records that could be potentially beneficial. I would suggest that a person should allow for a two to three

51

week time frame. Obviously this would allow more time for research, but it would also improve the quality of the search. With that kind of time, source documents for Fort Washita could be uncovered and useful historical documentation added. This was the only fort in the study for which I could not produce reliable documentation. Also, I would suggest taking a larger quantity of mortar samples from the area. This would allow the comparison of the CaCO3 from the mortar with another sample with a known standard. A comparison such as this would be a welcomed addition to this research.

#### REFERENCES

#### Boynton, Robert S.

1980 Chemistry and Technology of Lime and Limestone. Wiley Press.

#### Bradley, Betty H.

1999 *The Works: The Industrial Architecture of the United States.* Oxford University Press, 1999.

#### Cliver, E.B.

1974 Tests for Analysis of Mortar Samples. Bulletin of the Association for Preservation Technology VI(1): 68-73

#### Elliot, Cecil D.

1992 Technics and Architecture: The Development of Materials and Systems for Buildings. The MIT Press.

#### Franzini, Marco, Leoni, Leonardo, Lezzerini, Marco.

2000 A Procedure for determining the Chemical Composition of Binder and Aggregate in Ancient Mortars: its Application to Mortars from some Medieval Buildings in Pisa. *Journal of Cultural Heritage 1*: 365-373.

#### Fowler, David

2009 Personal Correspondence

#### Gordon, Robert B., Malone, Patrick M.

1994 *The Texture of Industry: An Archaeological View of the Industrialization of North America.* Oxford University Press.

# Guluc, Ahmet and Tulun, Tulay

1997 Physico-Chemical and Petrographical Studies of Old Mortars and Plasters of Anatolia. *Cement and Concrete Research* 27(2): 227-234.

Howe, Dennis E.

2007 An Archaeological Survey of the Whiteport Cement Works. *The Journal of the Society for Industrial Archeology 33(1).* 

#### Huffman, George G.

1958 Geology of the Flanks of the Ozark Uplift: Oklahoma Geological Survey Bulletin 77: 281

#### Jedrzejewska, H.

1960 Old Mortars in Poland: A New Method of Investigation. *Studies in Conservation:* 5(4): 132-138

#### Krotzer, Dorothy S., Walsh, John T.

2009 Analyzing Mortars and Stuccos at the College of Charleston: A Comprehensive Approach, *APT Bulletin 40(1)*: 41-48.

#### Puckette, James O.

2010 Personal Correspondence

#### Reedy, Chandra L.

1994 Thin-Section Petrography in Studies of Cultural Materials. Journal of the American Institute for Conservation 33(2): 115-129.

Riccardi, M.P., Duminuco, P., Tomasi, C., and Ferloni, P.

1998 Thermal, microscopic, and X-ray Diffraction Studies of Some Ancient Mortars. *Thermochimica Acta 321: 207-214* 

#### Ries, H. and Watson, Thomas L. 1936 Engineering Geology. John Wiley & Sons, New York.

#### Loeppert, R.H., Suarez, Donald L.

1996 Methods of Soil Analysis Part 3: Chemical Methods, edited by D.L. Sparks, A.L. Page, P.A. Helmke, R.H. Loeppert, P.N. Soltanpour, M.A. Tabatabai, C.T. Johnston, M.E. Sumner pp. 440-444. Soil Science Society of America, Inc., American Society of Agronomy, Inc., Madison, Wisconsin.

#### McCauley, Marissa

2008 March, 28. *How Were The Egyptian Pyramids Built?* from http://www. Sciencedaily.com/releases/2008/03/080328104302.htm; accessed 1 March, 2011.

#### Mckee, Harley J.

1961 Canvass White and Natural Cement, 1818-1834. Journal of the Society of Architectural Historians 20(4): 194-1971. Rowland, Ingrid D. and Howe, Thomas Noble

1999 Vitruvius: Ten Books of Architecture. Cambridge University Press.

Scholle, Peter A.

1979 Memoir 28: A Color Illustrated Guide to Constituents, Textures, Cements, and Porosities of Sandstones and Associated Rocks. The American Association of Petroleum Geologists.

#### Schnabel, Lorraine.

2009 Mortar Analysis Part2: Analytical Methods *APT Bulletin 40*(2): 1-7.

#### Starr, Chester G.

1991 A History of the Ancient World. Oxford University Press.

Stewart, John, Moore, James.

1982 Chemical Techniques of Historic Mortar Analysis. Bulletin of the Association for Preservation Technology 14(1): 11-16.

United States Geologic Survey 1960 Geologic Map of the United States. 1 sheet

Varas, M.J., de Buergo, Alvarez M., Fort, R.

2005 Natural Cement as the Precursor of Portland Cement: Methodology for its Identification. *Cement and Concrete Research 35*: 2055-2065.

#### VITA

#### PATRICK DEE HURLEY

#### Candidate for the Degree of

#### Master of Science

# Thesis: A MINERALOGICAL AND RESOURCE PROCUREMENT STUDY OF PRE-CIVIL WAR FORT CONSTRUCTION USING STONE AND MORTAR IN OKLAHOMA

Major Field: PLANT AND SOIL SCIENCE

Biographical: Born and raised in Enid, OK 9 May, 1982.

Education: Graduated Enid High School May, 2001 with honors and certificate of distinction. Accepted Presidential Nomination to the United States Military Academy at West Point May, 2002. Received Bachelor of Science in Horticulture, Turf Management option May, 2008.

Completed the requirements for the Master of Science in your Plant and Soil Science at Oklahoma State University, Stillwater, Oklahoma in May, 2013.

Completed the requirements for the Bachelor of Science in Horticulture at Oklahoma State University, Stillwater, Oklahoma in May, 2008.

Experience: Spent last five years working at the United Parcel Service, recently promoted to center manager.

Professional Memberships: N/A