

**A MAMMOTH OF A PROJECT:  
THE CONSERVATION OF A COLUMBIAN MAMMOTH**

A Thesis

by

SHANNA LAREA DANIEL

Submitted to the Office of Graduate Studies of  
Texas A&M University  
in partial fulfillment of the requirements for the degree of

MASTER OF ARTS

May 2007

Major Subject: Anthropology

**A MAMMOTH OF A PROJECT:  
THE CONSERVATION OF A COLUMBIAN MAMMOTH**

A Thesis

by

SHANNA LAREA DANIEL

Submitted to the Office of Graduate Studies of  
Texas A&M University  
in partial fulfillment of the requirements for the degree of

MASTER OF ARTS

Approved by:

Chair of Committee,  
Committee Members

Head of Department,

Charles Wayne Smith  
Michael Waters  
Darryl de Ruiter  
David Carlson

May 2007

Major Subject: Anthropology

## **ABSTRACT**

A Mammoth of a Project:

The Conservation of a Columbian Mammoth. (May 2007)

Shanna LaRea Daniel, B.A., Stephen F. Austin State University

Chair of Advisory Committee: Dr. Charles Wayne Smith

This thesis concentrates on discovering the best consolidant or consolidants for stabilizing a Columbian mammoth's sub-fossilized mandibles, a distal femur, an ulna, a radius, and a tooth. It was recovered from a wet, sandy gravel pit owned by the Vernor Family located in Clute, Texas. Based on thermoluminescence dating, the mammoth dates to around 66,000 years ago. The bones are fragile and unstable. They retain a minute amount of organic material (collagen) and hydroxyapatite, but not enough to retain any structural support.

Experiments and analyses were conducted on various bone samples to compare each of the following consolidants' properties. The consolidants examined were silicone oil, polyvinyl acetate (PVA) with viscosity of 25, Acryloid B-72, Butvar 98, Starbond EM-02, methyltrimethoxysilane (MTMS), Paleo-bond, and Rhoplex (Primal) WS24.

Stability, strength, and appearance were evaluated by measurable observations. The Scanning Electron Microscope (SEM) and the Environmental Scanning Electron Microscope (ESEM) at the Microscopy and Imaging Center at Texas A&M University were used to map penetration of these consolidants. SEM was utilized for both imaging and energy-dispersive x-ray spectroscopy (EDS) to examine the presence and absence of

certain elements. ESEM was used to view consolidants at the microscopic level to further examine the bonding between the consolidant and the bone's cellular structure.

By examining and testing all the consolidants, methyltrimethoxysilane (MTMS) was chosen to stabilize the ulna, radius, left and right mandibles, distal femur, and tooth. This research opened new avenues to different methods in preserving sub-fossilized bone and broadens our understanding of bone conservation.

## ACKNOWLEDGEMENTS

I would like to thank my committee chair, Dr. Wayne Smith, and my committee members, Dr. Michael Waters and Dr. Darryl de Ruiter, for their guidance and support throughout the course of my research.

Thanks also to the Center for Studies of the First Americans, Archaeological Preservation Research Lab, Conservation Research Lab, Microscopy and Image Center at Texas A&M University, and the Vernor Family for giving me the opportunity and means to conduct my research. I would like to also acknowledge the Hot Springs Mammoth Site staff and the University of Texas Vertebrate Paleontology Lab for the assistance in my research. I also want to extend my gratitude to Dr. Helen Dewolf for her guidance and support, as well as to my friends, colleagues, and the department's faculty and staff for assisting me during my time at Texas A&M University.

Finally, thanks to my mother, Kellie Richardson, and my father, Benny Richardson, for their love and encouragement, and to my husband, Joshua Daniel, for his love, patience, and undying support.

## TABLE OF CONTENTS

	Page
ABSTRACT.....	iii
ACKNOWLEDGEMENTS.....	v
TABLE OF CONTENTS.....	vi
LIST OF FIGURES.....	viii
LIST OF TABLES.....	xi
 CHAPTER	
I     INTRODUCTION – RAISING THE MAMMOTH	
Introduction.....	1
History of Mammoths.....	4
Vernor Site Columbian Mammoth.....	5
Removal of the Columbian Mammoth.....	9
Removal and Observation of the Columbian Mammoth before Conservation.....	16
II    COMPARISON OF BONE CONSOLIDANTS	
Introduction.....	21
Quantitative Experiments.....	26
Qualitative Experiments.....	38
Experiments with MTMS Treated Bone.....	45
III   EXPERIMENTAL RESULTS AND DISCUSSION	
Introduction.....	49
Results and Discussion.....	49
IV    AN INNOVATIVE METHOD FOR BONE CONSERVATION	
Introduction.....	51
Radius.....	51
Left Mandible.....	61
Right Mandible.....	68

CHAPTER	Page
Ulna.....	71
Distal Femur.....	74
Tooth.....	76
V CONCLUSION	
Summary and Conclusion.....	81
REFERENCES CITED.....	84
VITA.....	88

## LIST OF FIGURES

FIGURE	Page
1.1 Site overview.....	2
1.2 Mammoth orientation.....	6
1.3 Field conservation.....	10
1.4 Pedestal left mandible, ulna, and radius on site.....	12
1.5 Casting methods.....	12
1.6 Casting results.....	15
1.7 Applying heat to speed up the drying process of the casts.....	15
1.8 Moving casts into vehicles to be transported to Texas A&M University.....	16
1.9 Cast of right mandible.....	17
1.10 Manganese encrustation on the right mandible.....	18
1.11 The radius, ulna, and left mandible after removing the cast and most of the soil.....	20
2.1 The greenish discoloration that can occur when using EM-02 cyanoacrylate glue on the radius bone.....	35
2.2 Energy-dispersive x-ray spectroscopy (EDS) plot of control bone before conservation.....	39
2.3 Environmental scanning electron microscopy (ESEM).....	40
2.4 Energy-dispersive x-ray spectroscopy (EDS) plot of silicone oil treated bone.....	41
2.5 Energy-dispersive x-ray spectroscopy (EDS) plot of methyltrimethoxysilane coated bone sample.....	42
2.6 Energy-dispersive x-ray spectroscopy (EDS).....	43



FIGURE	Page
4.1 The radius after removing the cast.....	52
4.2 Showing the methodology of conserving the Columbian Mammoth using methyltrimethoxysilane.....	53
4.3 Marking the radius before taking it apart piece by piece.....	54
4.4 Removing the pieces from the radius for cleaning and reconstruction.....	55
4.5 Air scribing the distal end of the radius.....	56
4.6 The results after air scribing the distal end of the radius.....	56
4.7 Radius.....	58
4.8 Conserved radius.....	60
4.9 Left mandible.....	62
4.10 Marking the top portion of the left mandible before disassembling the pieces.....	62
4.11 The first stages of cleaning after removing the marked pieces from the top portion of the left mandible.....	63
4.12 The left mandible after gluing but before adding a mold to help stabilize the upper jaw.....	65
4.13 Left mandible reconstruction.....	66
4.14 Conserved left mandible.....	67
4.15 The right mandible's appearance while in the process of cleaning.....	69
4.16 Conserved right mandible.....	70
4.17 Photograph of the discoloration caused by acidic tissue paper and aluminum foil.....	71

FIGURES	Page
4.18 After cleaning the ulna, there was still noticeable discoloration and encrustation on the surface.....	72
4.19 Conserved ulna.....	73
4.20 Photography of distal femur before conservation.....	74
4.21 Conserved distal femur.....	75
4.22 Tooth.....	77
4.23 Conserved tooth.....	79

**LIST OF TABLES**

TABLES		Page
1	The before and after weight, stoichiometry length, width, and depth of each sample to evaluate any changes, such as distortion or shrinkage, that occurred during conservation.....	25
2	The quantitative measurements of penetration, strength, stability, and color of each bone after conservation.....	26
3	The before and after weight of conserving already MTMS treated bone.....	45
4	Observing and measuring the penetration, stability, and strength of each consolidant on MTMS treated bone.....	46

## CHAPTER 1

### INTRODUCTION: RAISING THE MAMMOTH

#### Introduction

A disarticulated Columbian mammoth was recovered by the late Dr. Robson Bonnicksen, Dr. Michael Waters, Dr. Darryl de Ruiter and students from the Center for the Study of the First Americans (CSFA) at Texas A&M University in conjunction with the Brazosport Archaeological Society. A six-week excavation took place in a commercial sandy gravel pit 18 meters below ground level owned by the Vernor Family in Clute, Texas (Figure 1.1 a and b). Two tusks, a left and right mandible, a distal femur, an ulna, a radius, and a tooth were recovered (Figure 1.2). Another less complete tusk was discovered east of the excavation. Based on thermoluminescence dating, the mammoth dates around 66,000 years ago. Its fragile and unstable skeletal remains retain a minute amount of organic material (collagen) and hydroxyapatite, but not enough to retain any structural support.

---

This thesis follows the style of *American Antiquity*.



(a)



(b)

**Figure 1.1. Site overview. a) Overview of the sandy gravel pit in Clute, Texas. b) Overview of the Columbian mammoth site.**

This thesis concentrates on discovering the best consolidant or consolidants for stabilizing this Columbian mammoth's remains. Experiments and analyses were conducted on various bone samples to compare each of the following consolidants' properties. The consolidants examined were silicone oil, polyvinyl acetate (PVA) with viscosity of 25, Acryloid B-72, Butvar 98, Starbond EM-02, methyltrimethoxysilane (MTMS), Paleo-bond, and Rhoplex (Primal) WS24.

Stability, strength, and appearance were evaluated by measurable observations. The Scanning Electron Microscope (SEM) and the Environmental Scanning Electron Microscope (ESEM) at the Microscopy and Imaging Center at Texas A&M University were utilized to map penetration of these consolidants. SEM was used for both imaging and energy-dispersive x-ray spectroscopy (EDS) to examine the presence and absence of certain elements. ESEM was used to view consolidants at the microscopic level to further examine the bonding between the consolidant and the bone's cellular structure. As part of the evaluation process, all treated samples were judged on their aesthetic appeal. It is important that consolidated bone look as natural as possible for future studies and viewing.

The principal of reversibility is a very important aspect to consider while examining consolidants for bone conservation. It is a desirable trait, but complete reversibility can never truly be achieved (Cronyn 1990: 9). Thus, retreatability may a more suitable term when reviewing different consolidation properties (Smith 2003: 5).

By examining and testing all the consolidants used for bone conservation, one was chosen to stabilize the ulna, radius, left and right mandible, distal femur, and tooth.

This research will open new avenues in preserving bone as well as broaden our understanding of bone conservation.

### **History of Mammoths**

Europe's earliest mammoths, *Mammuthus meridionalis*, first appeared between 3 million and 2.5 million years ago. They were the direct descendants of a tropical species that migrated out of Africa (Lister and Bahn 1994:12). *M. meridionalis* began to migrate into the New World during the Pleistocene Epoch, which began approximately 1.7 million years ago. This epoch is commonly known as the Ice Age because of its episodes of extremely cold climates leading to glacial conditions. During some of the coldest periods, the polar ice sheets expanded allowing the sea level to drop causing land bridges to form between Asia and Alaska. *M. meridionalis* began their migration eastward across this land bridge into the New World approximately 1.5 million years ago (Lister and Bahn 1994:12; Haynes 1991:5-6). This species was the ancestral mammoth from which two separate lines evolved: *Mammuthus trogontherii* (Steppe Mammoth) and *Mammuthus columbi* (Columbian mammoth) (Lister and Bahn 1994:12).

The species *M. trogontherii* evolved into *M. primigenius*, commonly referred to as the woolly mammoth in Europe and North America (Lister and Bahn 1994:14-15). While the woolly mammoth mainly occupied the northern region of the New World, the Columbian mammoth ventured into the southern regions of Canada, throughout the United States, and as far south as Nicaragua and Honduras. They stood around 13 feet at the shoulder and weighed about 10 tons (Lister and Bahn 1994:14-15; Agenbroad

1999:1). The Columbian mammoths' appearance was like the modern large Asian elephants with sparse hair covering its body (Agenbroad 1999:1).

Mammoths thrived over the vast territory of Europe, Asia, and North and South America for millions of years. Around 10,000 years ago at the start of the Holocene Epoch, mammoths became extinct. Various theories have been proposed on the extinction of the mammoths, but the influence of humans and extreme changes in the environment were key factors in their demise (Lister and Bahn 1994; Haynes 1991).

### **Vernor Site Columbian Mammoth**

According to Dr. Ernie Lundelius from the University of Texas in Austin, this particular Columbian mammoth died at an old age. The right and left mandibles both show evidence that the mammoth was using its last molar. After a mammoth loses all its teeth, it is unable to sustain the diet which it needs to survive.

Columbian mammoth teeth indicate that they were predominantly grazers. The molars have a tight cellular structure and multiple layers of enamel and dentine to help retain its integrity (Haynes 1991: 6-7). Just like modern elephants, mammoths grew six sets of teeth over their lifespan. Molars would eventually wear through and new molars would slowly push their way out, replacing the old teeth. Eventually the last set of teeth would wear away, leading to the mammoth's death by starvation.





**Figure 1.2. Mammoth orientation. Two tusks, a left and right mandible, distal femur, ulna, radius, and maxilla of a disarticulated Columbian mammoth at the Vernor Site.**

All bones, whether human or animal, are made up of components of both mineral (calcium phosphate with various carbonates and fluoride) and organic compounds (collagen and ossein) (Davis 1987; Hedges 1987; Smith 2003; Storch 1983). The organic ossein begins to decompose by hydrolysis after the bone is buried. The rate of deterioration depends on its environment. Acidic soils increase the rate of deterioration while alkaline soils decrease the degradation process. In waterlogged sites, bones are reduced to a sponge-like material, while in arid sites, the bones become dry and brittle.

In some cases, bones become fossilized when ossein is replaced with silica and mineral salts. Fossilization occurs when mineral matter such as silica replaces the organic materials within the bone (Schiffer 1987: 184)

After examining the mammoth's bones, it was concluded that they are sub-fossilized, which is a primary trait of bone from the Pleistocene and early Holocene Epochs. Sub-fossilized bone is "characterized by a loss of the proteinaceous framework in some degree with no consequent mineralization from geologic sources in the burial environment. Both the cortex and the medulla are affected by the loss of proteinaceous structure, which causes a diminution of the elasticity and strength of the bone" (Shelton and Johnson 1995: 60). Furthermore, the use of acidic or caustic materials during casting or storage may cause irreversible damage to sub-fossilized bone (Shelton and Johnson 1995: 60). This is important to consider when dealing with sub-fossilized bone.

The mammoth's remains were well preserved due to various environmental factors. To understand what caused the deterioration or preservation of the mammoth, a brief look at the taphonomy was essential. Taphonomy includes the analysis of the natural and cultural processes surrounding the deposition of the bones. Natural processes involve chemical, biological, and physical agents. Cultural processes involve deliberate and/or accidental alterations of the bones by humans (Schiffer 1987: 148). In this particular case, there were no artifacts associated with the mammoth, nor was there any evidence of human alteration on the bones. Therefore, only the natural environmental processes affected the remains.

There are various chemical agents which could have affected the composition of its remains. The soil's pH level can chemically alter the structure. Soil contains reactive compounds (acids and bases) that contribute to the deterioration process (Schiffer 1987: 150). The soil at the Vernor site has a neutral or more alkaline pH level, aiding in the preservation of its remains. Fossilization is another chemical process. As mentioned earlier, the mammoth's bones are sub-fossilized.

Physical agents can either help reduce or advance the bone's deterioration. Soil deposits, water movement, natural disasters, and sunlight are known as physical agents (Schiffer 1987: 149). The deposition of sediments can cause pressure on buried bones and cause distortion. On the other hand, it can lead to better preservation of the bones. Sediments can protect bone from other physical or biological agents, such as sunlight and gnawing predators. When exposed to the surface, bones deteriorate with fluctuating temperatures, weathering, and sunlight. Buried bones are susceptible to ground water or other water environments, causing them to become waterlogged. The Columbian mammoth was waterlogged upon discovery. When a specimen is taken from a waterlogged environment without remaining wet or having a bulking agent to replace the water, the organic matrix loses strength and stability. This leads to cracks, fractures, and exfoliation, as well as increasing the rate of deterioration to the point of losing the artifact or specimen.

Biological agents play an important role in understanding the bone's modification. When dead organisms are first exposed to the environment, bacteria cause the organism to decay. This process allows other animals to consume or modify the

bones. Animals, such as beetles, ants, flies, and termites, consume the bone's organic components. Mammalian or avian scavengers also gnaw on bones causing further modifications. More than likely, some of these factors did have an effect on the Columbian mammoth, but there was no observable evidence. By understanding the processes surrounding an artifact or specimen, correct conservation methods can be utilized to stabilize the artifact, in this case a Columbian mammoth

### **Removal of the Columbian Mammoth**

This section focuses on the preparation techniques, casting methods, and removal of the Columbian Mammoth from the site. This is a crucial step in the conservation process. While excavating, the bones and tusk were kept wet to ensure that they did not dry out. There was an attempt to conserve the remains with a mixture of 95% denatured alcohol and 5% Acryloid B-72. This solution was topically applied, but the Acryloid B-72 began to react to the water within the bones (Figure 1.3 a and b). Also, certain areas contained manganese encrustation, which further restricted the solution's penetration into the bones' matrix.



(a)



(b)

**Figure 1.3. Field conservation. a) Dr. Darryl de Ruiter conserving a bone using Acryloid B-72 on site. B) The results after conserving in the field.**

Preparations were made to begin casting and removing the bones and tooth. To begin the casting process, the bones were pedestaled on the sandy, clay soil for support. The right mandible was raised by itself. The left mandible, ulna, and radius were lifted together (Figure 1.4). The casting process began by adding wet tissue paper around the bones followed by a layer of aluminum foil. Duct tape was added to the top of the aluminum foil to secure it. Afterwards, bubble wrap secured with tape was placed on top to protect the bones from the plaster. Burlap straps of various lengths were cut and immersed in water. After they were fully saturated, they were placed in plaster of paris. The crew began to cast the right and left mandible, radius, and ulna (Figure 1.5 a-e). The casts were left to dry before removal. Due to wet weather conditions, the plaster of paris did not harden completely (Figure 1.6). Heaters were rented to speed up the drying process; after several hours, the plaster was dry enough for the bones and tusk to be removed (Figure 1.7). Using machinery from the Vernor Material and Equipment Company, all the casts were removed and transported to Texas A&M University (Figure 1.8).



**Figure 1.4. Pedestal left mandible, ulna, and radius on site.**



**(a)**

**Figure 1.5. Casting methods. a) Placing wet tissue paper on tusk and bones**



(b)



(c)

**Figure 1.5. Continue. b) Applying aluminum foil over the tissue paper. c) Placing bubble wrap over aluminum foil.**





(d)



(e)

**Figure 1.5. Continue. d) Applying burlap straps soaked in plaster of paris. e) Final cast of the bones.**



**Figure 1.6. Cast results. After wet weather conditions over the weekend, the cast of both the bones and tusk were destroyed.**



**Figure 1.7. Applying heat to speed up the drying process of the casts.**



**Figure 1.8. Moving casts into vehicles to be transported to Texas A&M University.**

### **Removal and Observation of the Columbian Mammoth before Conservation**

The bones and tusk were transported to the Archaeological Preservation Research Lab, directed by Dr. Wayne Smith, at Texas A&M University. To ensure the bones did not dry out, wet towels were applied to the cast and around openings. This allowed moisture to remain in the casts.

The right mandible cast was the first to be unwrapped using a razor blade (Figure 1.9). While removing the soil and the cast, the right mandible's instability was evident. One piece of the mandible fell off due to one of two reasons; it was already broken or cracked and the soil was holding the piece together, or it was cracked or broken during the removal or transportation. There were observable manganese encrustations and fragile areas on the mandible's surface (Figure 1.10). Most of the soil was removed without harming the mandible, but it was left in the cast for stability and support.



**Figure 1.9. Cast of the right mandible.**



**Figure 1.10. Manganese encrustation on the right mandible.**

Dr. Boyd, professor at Texas A&M University Veterinary School, graciously allowed CSFA to use their circular saw to remove the larger-size cast. By using this saw, the left mandible, ulna, and radius's cast was removed. New stress fractures and cracks were observed causing the left mandible and the radius to break during their removal. Despite the hard work in the field, the cast did not set well, allowing the soil and bones to move during their transportation and removal. Furthermore, the left mandible, radius, and ulna cast had little soil supporting them. It would have been in the best interest of these artifacts to cast each bone separately or build a rigid framework underneath and/or around the cast.

Another reason why new cracks and stress fractures were observed was due to the flux in relative humidity (RH), temperature, and unmonitored drying. Warping, splitting, cracking, and exfoliation were observed while slowly drying out waterlogged bone at the Windover Site in Florida. This suggested the bone's structural strength was weakened "due to the loss of organic material, the breakage of protein chains, or the

weakening of the bonds between the organic and inorganic constituents” (Stone et al. 1990:180).

Upon removing the left mandible, ulna, and radius, a rusty orange discoloration was noticeable (Figure 1.11). The discoloration was due to the combination of acidic tissue paper and aluminum foil during the casting process. Most of the disfigurement occurred in places where the manganese encrustations were located. Since the tissue paper contained bleach, it reacted to the encrustation causing the tissue paper to discolor. One researcher mentioned using wet-acid free tissue paper over the entire artifact before proceeding with plaster of paris (Cronyn 1990: 46). After researching casting techniques, it is my opinion that there is a need to revise certain casting methods, especially when waterlogged artifacts can not be transported in water. Steps need to be taken in the field to ensure the artifact is in a stable environment before being sent to the laboratory. After observing the mammoth’s fragile bones, experiments were conducted with different bone consolidants to see what consolidant would be best to stabilize them.



**Figure 1.11. The radius, ulna, and left mandible after removing the cast and most of the soil.**

## CHAPTER II

### COMPARISON OF BONE CONSOLIDANTS

#### **Introduction**

Throughout the 20<sup>th</sup> century, archaeologists and conservators have used organic polymers to conserve bone. But, only a few polymer solutions have been utilized, despite the large variety of consolidants available in today's market. The most commonly used consolidants are polyvinyl acetate (PVA), polyvinyl acetate emulsions, natural resins, acrylic emulsions, and acrylic colloidal dispersions (cf. Cronyn 1990; Hamilton 2000; Johnson 1994; McCarty 2002; O'Connor 1987; Potapova 2005; Rainey 2004; Sease 2003; Shelton and Johnson 1995; Singley 1988; Wheatcroft 1994). Of the many studies covering bone conservation, one study (Johnson 1994) gives a thorough overview on consolidant properties and their uses. It is a good synopsis on the past uses of consolidants, such as polyvinyl acetate emulsions like Elmer's Glue-All and nitrocellulose resins such as Duco cement. Due to the unknown properties of these proprietary consolidants and their non-stable nature within organic artifacts, they have become outdated and been replaced by more reliable and stable polymer consolidants. This study reviews and discusses all the properties, uses, and analyses on current bone consolidants used in today's conservation labs.

One of the things not substantially researched in bone conservation is the use of cyanoacrylates (superglues). In recent years, the use of superglues has increased, not just as an adhesive, but as a consolidant for bone. Potapova (2005) and Rainey (2004) mention the growing use of cyanoacrylates in their labs. Cyanoacrylates' growing



popularity can be characterized by their quick setting time and strength, but there is still little known about its longevity (McCarty 2002:5). Recent preliminary studies have shown that cyanoacrylates are made up of different esters (ethyl, methyl, propyl, and butyl), which can have an adverse effect on the degradation of the fossil material (Down and Kaminska 2006). Nevertheless, there is still the call for future studies on cyanoacrylates.

In the past few years, conservation methods were developed for conserving waterlogged artifacts using acrylic emulsions and dispersions, polyvinyl acetate emulsions and resins, polyethylene glycol (PEG), and silicone oil (cf. Bronstein 1981; Cronyn 1990; Singley 1988; Smith 2003; Stone et al. 1990; Wheatcroft 1994). Emulsions and dispersions are water-based consolidants that allow waterlogged artifacts to be placed into the solution without going through a dehydration process. Polyethylene glycol (PEG), known as Carbowax, is a popular consolidant for waterlogged artifacts. *The Preservation and Conservation of Waterlogged Bone from the Windover Site, Florida: A Comparison of Methods* (Stone et al. 1990), discusses the pros and cons of using acrylic emulsions/dispersions and PEG on waterlogged bone. The two consolidants being tested were PEG and Rhoplex. The consensus of post-treatment analysis indicates that Rhoplex appears to be the best option for conserving bone. Over the years, PEG has shown to be unstable in certain environmental conditions and adds little structural support to any organic artifact over time.

A new technology was developed using Passivation Polymers to conserve waterlogged artifacts. In *Archaeological Conservation Using Polymers* (Smith 2003), it

discusses conserving ivory and bone using this silicone oil based technology. To initiate this process, a period of dehydration is needed to replace water with an organic solvent, either ethanol or acetone. Using a solution consisting of silicone oil with a crosslinker (methyltrimethoxysilane) and a catalyst (dibutyltin diacetate or TPT Tinacetate) to finalize the process, the outcome gives once fragile waterlogged bone structural stabilization in any environmental condition. This method is coined the “Next Generation Process for Biological Specimen Preservation” (Glover et al. 2003:1). The crosslinker MTMS is also a polymer that can be used to conserve various organic and non-organic artifacts (cf. Charole et al. 1984; Smith 2003, 2006). There is no known literature about using MTMS for conserving bone.

Bone conservation involves a consolidant to permeate deep into the cellular structure. Consolidants are resins available in pure form or emulsions which dissolve in a solvent of water, alcohol, or acetone. (McCarty 2002:1). The two chemical compositions best suited for bone are polymers and cyanoacrylates. Polymers are very large molecules which are made by linking together one or more small molecules called monomers (Wheatcroft 1994:25). Most polymers in conservation are applied in a liquid state, which eventually evaporates after application, leaving strands of polymer chains.

Another type of consolidant used for bone is cyanoacrylates. Cyanoacrylate adhesives are acrylate monomers with the chemical formula  $C_5H_5NO_2.R$ . In this formula, R is a side-group of either methyl ( $-CH_3$ ), ethyl ( $-C_2H_5$ ), or butyl ( $-C_4H_9$ ). The smaller the side group, such as methyl, the stronger and faster the curing time will be for consolidating. Whereas the larger the side group, such as butyl, the weaker the bond and

the slower the curing time will be for consolidating (Wheatcroft 1994: 56). Preliminary studies have shown that a larger side group in a cyanoacrylate chemical formula, such as butyl, has the slowest degradation rate on bone (Down and Kaminska 2006: 524).

After reviewing the literature regarding bone conservation, experiments and analyses were conducted on various bone samples to compare each of the following consolidants' properties. They were silicone oil, polyvinyl acetate (PVA) with a viscosity of 25, Acryloid B-72, Butvar 98, Starbond EM-02, methyltrimethoxysilane (MTMS), Paleo-bond, and Rhoplex (Primal) WS24.

Stability, strength, and aesthetic appeal were evaluated by quantitative observations. It is important that consolidated bone look as natural as possible for future studies and viewing. The principle of reversibility is a very important aspect to consider before conserving. As mentioned in Chapter I, reversibility is a desirable characteristic, but cannot be completely achieved. Scanning Electron Microscope (SEM) and Environmental Scanning Electron Microscope (ESEM) at the Microscopy and Imaging Center at Texas A&M University were utilized to map penetration. After testing all the consolidants, one was chosen to stabilize the ulna, radius, left and right mandible, distal femur, and tooth.

Due to the time constraints and funding, only 10 samples could be tested. This chapter discusses in-depth the effects of each consolidant as a conservation agent for bone. Each bone sample was traced, weighed, and measured before and after conservation. In addition, the bone's color was documented using the Munsell Soil Chart Book. After conserving each bone sample, evaluations were conducted to see how

well the treatment worked. Procedures taken for each bone sample is discussed below, beginning with the control. Tables 1 and 2 contains the before and after weight, width, length, depth measurements, stability, strength, color, and the consolidants' penetration into each bone sample.

Table 1. The before (B.) and after (A.) weight, stoichiometry length, width, and depth of each sample to evaluate any changes, such as distortion or shrinkage, that occurred during conservation.

<b>Consolidant</b>	<b>B. Weight</b>	<b>A. Weight</b>	<b>B. Length</b>	<b>A. Length</b>	<b>B. Width</b>	<b>A. Width</b>	<b>B. Thickness</b>	<b>A. Thickness</b>
Control	14 grams	14 grams	51.9 mm	51.9 mm	21.1 mm	21.1 mm	14.3 mm	14.3 mm
MTMS	6 grams	6 grams	39.6 mm	39.6 mm	13.0 mm	13.0 mm	11.5 mm	11.5 mm
MTMS with DBTDA	4 grams	4 grams	15.1 mm	15.1 mm	16.1 mm	16.1 mm	11.5 mm	11.5 mm
Silicone Oil	41 grams	41 grams	45.0 mm	45.0 mm	34.7 mm	34.7 mm	27.3 mm	25.2 mm
Polyvinyl Acetate (PVA)	8 grams	7 grams	41.3 mm	41 mm	13.8 mm	13.8 mm	13.6 mm	13.8 mm
Acryloid B-72	21 grams	19 grams	34.4 mm	34.1 mm	28.4 mm	28.3 mm	15.4 mm	15.4 mm
Polyvinyl butyral	5 grams	3 grams	59.2 mm	23.5 mm	21.2 mm	13.8 mm	19.9 mm	13.4 mm
Starbond EM-02	14 grams	12 grams	34.4 mm	34.4 mm	23.5 mm	23.5 mm	15.1 mm	15.1 mm
Paleo-bond	6 grams	5 grams	30.5, 7.1 mm	30.5, 7.1 mm	24.1 mm	24.1 mm	10.6 mm	10.6 m
Rhoplex WS-24	6 grams	6 grams	31.5 mm	31.5 mm	17.2 mm	17.2 mm	11.7 mm	11.7 mm

Table 2. The quantitative measurements of penetration, strength, stability, and color of each bone after conservation.

Consolidant	Penet.	Color	Stability	Strength	Comments
Control	0	10 YR 8/3 very pale brown	0	0	
MTMS	4	10 YR 8/3 very pale brown	2	2	does not change color; hardens porous structure
MTMS/DBTDA	4	10 YR 6/6 brownish yellow	2	2	minimal to no glossy film; darker color due to catalyst
Silicone Oil	4	10 YR 8/4 very pale brown	2	2	minimal to no glossy film; crystals observable on surface due to MTMS
Polyvinyl Acetate (PVA)	3	10 YR 8/3 very pale brown	2	2(1)	could yellow over time; 1 in strength for the bone's porous structure
Acryloid B-72	3	10 YR 8/3 very pale brown	2	2(1)	1 in strength for the bone's porous structure
Polyvinyl Butyral	3	10 YR 8/4 very pale brown	2	2	drastic color change after continuous application
Starbond EM-02	2	10 YR 8/4 very pale brown	2	2	glossy film, could turn green, sticky when too much was added
Paleo-bond	3	10 YR 6/3 pale brown	2	2	very sticky, leaves bone and soil a darker color
Rhoplex WS-24	2	10 YR 7/3 very pale brown	1	1	best for waterlogged bone, leaves glossy film

Stability (compare to control)

0 – Control  
1 – Moderate flaking  
2 – minimal to no flaking

Strength (under pressure)

0- Maximum crumbling  
1 – moderate crumbling  
2 – minimal or no crumbling

Penetration

0 – no penetration  
1 – surface only  
2 - surface/minimal  
3 – surface/moderate  
4 –surface/maximum

## Quantitative Experiments

### Control bone

The control bone weighed 14 grams. Its widest width was 51.9 mm, with a maximum length of 21.2 mm and thickness of 14.3 mm. The control crumbles and flakes under any type of pressure, which shows lack of structural durability. The color

of the sample was a very pale brown (10 YR 8/3 Munsell). All bone samples being treated were compared to this sample.

### MTMS

Methyltrimethoxysilane (MTMS) is a “silane cross linker selected from a phenyl, hydrogen, vinyl, or an alkyl group having one or two carbon atoms” (Klosowski 2004:5). It’s a functional polymer that creates a resin, which preserves the physical structure of the bone. There are three forms of resin in MTMS. The primary resin forms a methyl group with three siloxy bonds, for example Si – O – Si. The second resin forms two siloxy bonds or a double bond. The third resin forms methyl groups and hydroxyl bonds. The first resin is the strongest to bond with the bone’s chemical make-up. While, the third resin has the least amount of strength, and is usually vaporized out or left in a non-bonded state (Smith 2006: personal communication).

The bone sample treated with 100% MTMS had a before weight of 6 grams. The sample’s widest width was 13.0 mm with a maximum length of 39.6 mm and thickness of 11.5 mm. With a small brush, 100% MTMS was topically applied onto the bone. With each application, the bone exhibited an increase in hardness. Afterwards, the sample was left to dry. Its color after drying was very pale brown (10 YR 8/3 Munsell), which is the same as the control sample. There was no change in the weight, length, width, or thickness of the sample. There was minimal-to-no flaking, as well as minimal to no crumbling under pressure. This consolidant is not reversible.

### MTMS with a catalyst

This experiment looked at using the silane cross-linker, MTMS, with a catalyst to finalize the treatment. The catalyst, also known as a hardener, initiates and maintains the cross-linking process (Wheatcraft 1994: 37). The catalyst used in this experiment was dibutyltin diacetate (DBTDA).

This bone sample's before weight was 4 grams. The sample's widest width was 16.1 mm with a maximum length of 15.1 mm and a thickness of 11.5 mm. After topically applying 100% MTMS, the sample was placed in a sealed bag with a small aluminum dish containing DBTDA. The catalyst dissipates after a 24 hour period, so the procedure was repeated to complete the polymerization process.

After treatment, the bone sample's weight was 4 grams. Its length was 15.1 mm, 16 mm in width, and 11.5 mm in thickness. There was minimal to no flaking and minimal to no crumbling under pressure. The consolidant showed penetration through the surface and into the cancellous region. The color was brownish yellow (10 YR 6/6 Munsell). There was an observable difference in color when compared to the control. As it has been observed, MTMS alone does not cause this color change; it is the catalyst that gives the bone this brownish yellow hue. The disadvantage with using a catalyst with MTMS is the color change, as well as its irreversibility.

### Silicone oil

Passivation Polymer treatment using silicone oils is a conservation treatment developed by Drs. C. Wayne Smith and Donny Hamilton at Texas A&M University. This treatment uses a solution consisting of silicone oil with a cross-linker, MTMS. It

impregnates into the bones' voids and bonds to the cell walls to stabilize and prevent impurities from attaching and reacting with the bone.

The bone sample treated with silicone oil had a before weight of 41 grams. Its widest width was 34.7 mm with a maximum length of 45 mm and a thickness of 27.3 mm. Before treatment, the bone sample was placed in 100% acetone. Complete dehydration must be accomplished before immersing it in silicone oil. This allows a clear pathway for silicone oil to migrate into the bone's structure. Since the bone was already air dried, only the acetone step was needed. If the bone was still waterlogged, it would have had to go through several dehydration steps starting with 25% ethanol and 75% water. The ethanol would increase slowly until the solution reach 100% ethanol. Then, the sample would be place in a solution starting with 25% acetone and 75% ethanol. The amount of acetone would increase by increments of 25% until it reached 100% acetone. This would allow the water to slowly be removed from the bone without collapsing its cellular structure.

The bone sample was immersed in acetone for 24 hours. Afterwards, it was placed in a solution consisting of 97% silicone oil and 3% MTMS. The minimal amount of MTMS was chosen because this is the absolute minimum amount necessary to cross link the polymer. After further research, it is best to use a larger percent of cross-linker to ensure polymerization. It is suggested to use 70 to 80% silicone oil with a 20 to 30% by weight of methyltrimethoxysilane (Dewolf 2005: personal communication). The bone sample was removed after showing complete impregnation of the solution. The bone was allowed to drain unbounded silicone oil for several days. Afterwards, the



sample was placed in a sealed bag containing a small aluminum dish of TPT Tinacetate. This catalyst was chosen for its availability and is a stronger and more volatile catalyst than DBTDA (Smith 2005: personal communication). After 24 hours, a fresh dish of TPT Tinacetate catalyst was placed in the bag, allowing it to further catalyze.

After the treatment, the color was very pale brown (10 YR 8/4 Munsell). There was just a shade difference between the silicone oil's bone sample color and the control bone's color. There was no change in its weight, length, and width. The thickness changed from 27.3 mm to 26.9 mm. This change was due to the measurement accuracy of the examiner, but the difference is not drastic. There was minimal to no flaking and minimal to no crumbling under pressure. The sample showed the solution penetrated the surface and into the cancellous region. There was minimal to no glossy film on the surface. Due to the silane cross-linker, MTMS, there were observable crystals on the surface, but were removed by gently brushing the surface.

When compared to the control and other samples, silicone oil does not retain a glossy film or leave any sticky residue on the surface. Silicone oil is not temperature sensitive and is not affected by UV light. In addition, it does not change chemically in the presence of oxides. Another benefit of this treatment is its longevity. Tests show that this treatment can remain stable for 300 years, so further treatment or retreatment would not be needed for several generations. The only non-benefit of using silicone oil is its non-reversibility.

### Polyvinyl acetate or PVA

Polyvinyl acetate or PVA is commonly used in conserving archaeological artifacts. It is a long chain polymer consisting of carbon, hydrogen, and oxygen. It comes in different viscosities, but a lower viscosity is suggested to consolidate bone. Lower viscosities allow the solution to penetrate and spread more readily than higher viscosities. PVA can be removed using a strong solvent, but not all of the polymer chains can be completely removed from the artifact. So, it is best to say it is retreatable, but not reversible.

In this experiment, PVA with a viscosity of 25 was used because of its availability. A viscosity of 7 or 15 is considered good for consolidating organic materials as well. The bone sample conserved with PVA V25 weighed 8 grams before conservation. Its widest width was 34.7 mm with a maximum length of 41.3 mm and a thickness of 27.3 mm. A solution of 90% acetone and 10% PVA V25 was created. The bone sample was immersed in the solution for one week. Afterwards, there was some observable penetration, but the bone was soft due to the high amount of acetone within the solution. After the sample dried, the bone was stable enough to handle.

After conservation, the bone sample's color was very pale brown (10 YR 8/3 Munsell) with an observable shine on the surface. Its weight was 7 grams, length was 41 mm, width was 13.8 mm, and thickness was 13.8 mm. The loss of weight was attributed to the removal of water and oxygen molecules, which were replaced with acetone and PVA. There was observable penetration on the surface but only moderately into the sample. It exhibited minimal to no flaking. When pressure was applied, the surface had

minimal to no crumbling, but the porous interior showed moderate crumbling. This indicates the PVA solution did not migrate deep enough into the bone's structure to properly conserve.

### Acryloid B-72

Acryloid B-72 is one of the most commonly used materials in conservation. It is a co-polymer made of two kinds of monomers: ethyl methacrylate and methyl acrylate (Wheatcroft 1994: 30). It is a very stable polymer that can be used as a consolidant and an adhesive. It is less glossy than PVA and retains good flexibility. It does not yellow over time like PVA, and is resistant to water, alcohols, alkaline, acid, oils, and grease (Elder et. al 1997; Hamilton 2000). Acryloid B-72 is a good consolidant to use on bone discovered from desiccated sites. This consolidant can be removed using a strong solvent, but not all of the polymer chains can be completely removed from the artifact. So, it is best to say it is retreatable, but not reversible.

The bone sample conserved with Acryloid B-72 weighed 21 grams before conservation. The widest width was 28.4 mm with a maximum length of 34.4 mm and a thickness of 15.4 mm. A solution consisting of 90% acetone and 10% Acryloid B-72 was created. The bone sample was immersed in the solution for one week. After the bone was removed from the solution, there was observable penetration on the surface. The bone was soft due to the large amount of acetone in the solution. After drying, the bone was stable enough to handle.

The sample's color after conservation was very pale brown (10 YR 8/3 Munsell) with an observable glossy film on the surface. Its weight was 19 grams, length was 34.1

mm, width was 28.3 mm, and thickness was 15.4 mm. The loss of weight was attributed to the removal of water and oxygen molecules, which were replaced with acetone and Acryloid B-72. There was observable penetration on the surface and only moderately into the cancellous area. When pressure was applied, the surface had minimal to no crumbling, but the cancellous area had moderate crumbling. This indicates Acryloid B-72 solution did not penetrate into the bone's porous structure to conserve it properly.

#### Polyvinyl butyral

Polyvinyl butyral, known as Butvar 98, is a long chain polymer. It can be used as a consolidant and an adhesive. It comes in the form of a white, free flowing powder like material that is soluble in alcohols and acetone. According to Potapova, Head Conservator at the Hot Spring Mammoth Site in Hot Spring, South Dakota, Butvar 98 can change the bone's color drastically after continuous application (Potapova 2005: personal communication). Just like PVA and Acryloid B-72, Butvar 98 can be removed using a strong solvent, but not all polymer chains can be completely removed. So, it is best to say it is retreatable, but not reversible.

Before conservation, the weight of the bone sample treated with Butvar 98 was 5 grams. Its widest width was 13.5 mm with a maximum length of 23.5 mm and a thickness of 13.5 mm. A solution of 90% of denatured alcohol and 10% Butvar 98 was created. The sample was immersed in the solution for one week. After conservation, the color was very pale brown (10 YR 8/4 Munsell) with a weight of 3 grams. Its length was 23.5 mm, 13.8 mm in width, and 13.4 mm in thickness. Just like PVA and Acryloid B-72, the loss of weight was attributed to the replacement of water and oxygen

molecules with a solvent and Butvar 98. The solution penetrated the surface and into the sample only moderately. It had minimal to no flaking and minimal to no crumbling under pressure.

### Starbond EM-02

Starbond EM-02 is a cyanoacrylate, which is a type of superglue. It is made up of ethyl (-C<sub>2</sub>H<sub>5</sub>) cyanoacrylate, polymethyl methacrylate (Perspex or Plexiglas) and hydroquinone (Wheatcroft 1994: 55-56). This particular superglue has a very low viscosity that is able to penetrate into the cancellous regions of the bone. It is difficult to remove and may stain certain areas if used in great amounts. Furthermore, there has been no known testing on the longevity of cyanoacrylates.

The bone sample treated with Starbond EM-02 weighed 14 grams before treatment. The widest width was 23.5 mm with a maximum length of 34.4 mm and a thickness of 15.1 mm. Starbond EM-02 was topically applied onto the bone. When the bone began to look wet, the application process stopped. Then, a cyanoacrylate activator (catalyst) was sprayed once on the bottom and on the top. If an unnecessary amount of activator came into contact with the cyanoacrylate, it would turn the bone green (Rainey 2004: personal communication) (Figure 2.1). After the experiment, there was a greenish hue on the newspaper located under the bone but no discoloration was observed on the bone itself.



**Figure 2.1. The greenish discoloration (indicated by the red arrow) that can occur when using EM-02 cyanoacrylate glue on the radius bone (photography by Dr. Wayne Smith).**

After conservation, the weight of the sample was 12 grams. The length was 34.4 mm, width was 23.5 mm, and thickness was 15.1 mm. There was no distortion or shrinkage of the sample. The color of the treated bone was very pale brown (10 YR 8/4 Munsell). It had minimal to no flaking and minimal to no crumbling under pressure. The consolidant fully penetrated the surface, but only minimally into the cancellous area. A glossy film was observed on the surface. The sample was very sticky to the touch after treatment. This is an undesirable characteristic when dealing with bone.

### Paleo-bond

Paleo-bond is a cyanoacrylate. This particular glue is typically used with geological and paleontological materials (Elder et al 1997:1). It bonds well and has a quick curing time. These bonds can be reversed with a solvent but it is very difficult.

The bone sample treated with Paleo-bond weighed 6 grams before conservation. Its maximum length was 30.5 mm at one end and 7.1 mm at the other. The widest width was 24.1 mm with a thickness of 10.6 mm. Paleo-bond was topically applied until the bone appeared to look wet. Then, a catalyst was sprayed to complete the bonding process. The sample's color after conservation was pale brown (10 YR 6/3 Munsell), which gives the bone a darker appearance than the other treated samples. The weight was 5 grams. There were no changes in the length, width, or thickness, and there was no distortion or shrinkage. There was minimal to no flaking and minimal to no crumbling under pressure. Paleo-bond penetrated only the surface and moderately into the bone's matrix. The sample was sticky on the surface, which is an undesirable characteristic when handling bone.

### Rhoplex (Primal) WS-24

Rhoplex WS-24 is an acrylic emulsion. This particular consolidant dissolves in water instead of solvents. It has a finer particle size than PVA emulsions allowing the solution to disperse thoroughly into fragile bone (Johnson 1994: 17). Its composition consists of acrylic co-polymers, individual residual monomers, ammonia, and water. Like many other emulsion-based consolidants Rhoplex WS-24 may polymerize over time. The experiment on Rhoplex WS-24 could only be prepared on dry bone, so the

results will only indicate the reaction of acrylic emulsion on dry specimens. But, it is still ideal to use on damp or wet bone.

The bone sample treated with Rhoplex WS-24 weight before treatment was 6 grams. Its widest width was 17.2 mm with a maximum length of 31.5 mm and a thickness of 11.7 mm. A dilute solution was made with 1 part Rhoplex WS-24 to 10 parts water. The bone sample was immersed in the solution until off-gassing was complete. The sample was taken from the solution and dried. After treatment, the bone sample weighed 6 grams. The length was 31.5 mm, width was 17.2 mm, and the thickness was 11.9 mm. The color was very pale brown (10YR 7/3 Munsell). It exhibited moderate flaking and moderate crumbling under pressure. The consolidant penetrated the surface and minimally into the cancellous or porous region. There was no evidence of cracking or a glossy shine on the surface. Rhoplex WS-24 is not complete reversible, but can be retreated.

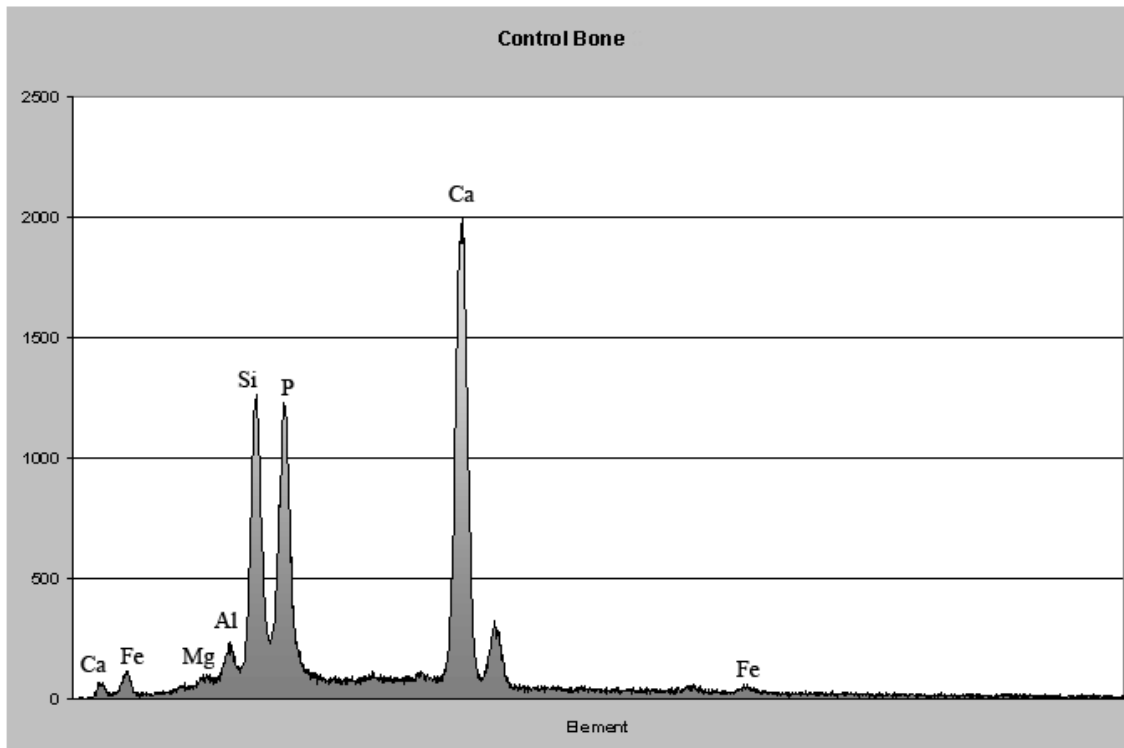
It is suggested Rhoplex WS-24 be used on wet or waterlogged bone. This consolidant would have been good to use as a temporary consolidant in the field. This water pre-mixed consolidant would have easily penetrated the surface and into the bones' matrix to help stabilize the bones during their removal.

Final analyses showed that all samples received some observable penetration and/or coating. However, silicone oil, methyltrimethoxysilane (MTMS), and MTMS with a catalyst showed the best penetration and stability. When compared to the control and other samples, these consolidants had the desirable characteristics for conserving bone.



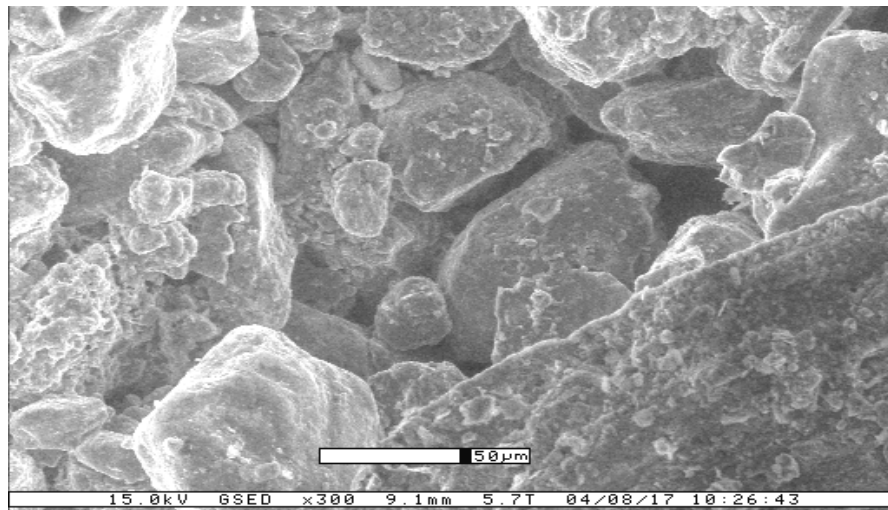
## Qualitative Experiments

SEM and ESEM were used to map penetration of both silicone oil and methyltrimethoxysilane. The SEM, JMS-6400, was used for both imaging and energy-dispersive x-ray spectroscopy (EDS) to examine the presence and absence of certain elements when compared to the control. Each sample was sectioned to view the bone's interior. The control sample's elemental analysis showed the normal chemical composition of bone, such as calcium (Ca), and phosphorus (P). Other elements, such as magnesium (Mg), iron (Fe), and silicon (Si) were found within the soil and ground water surrounding the Columbian Mammoth (City of Clute, Annual Drinking Water Quality Report, 2005) (Figure 2.2). Another explanation for the appearance of silicon is the bone's present state of mineralization. During mineralization, the organic components are replaced with mineral compounds, such as silicates and carbonates (Stone et al 1990:178). The aluminum (Al) is contributed by the base on which the sample was placed on during EDS testing.

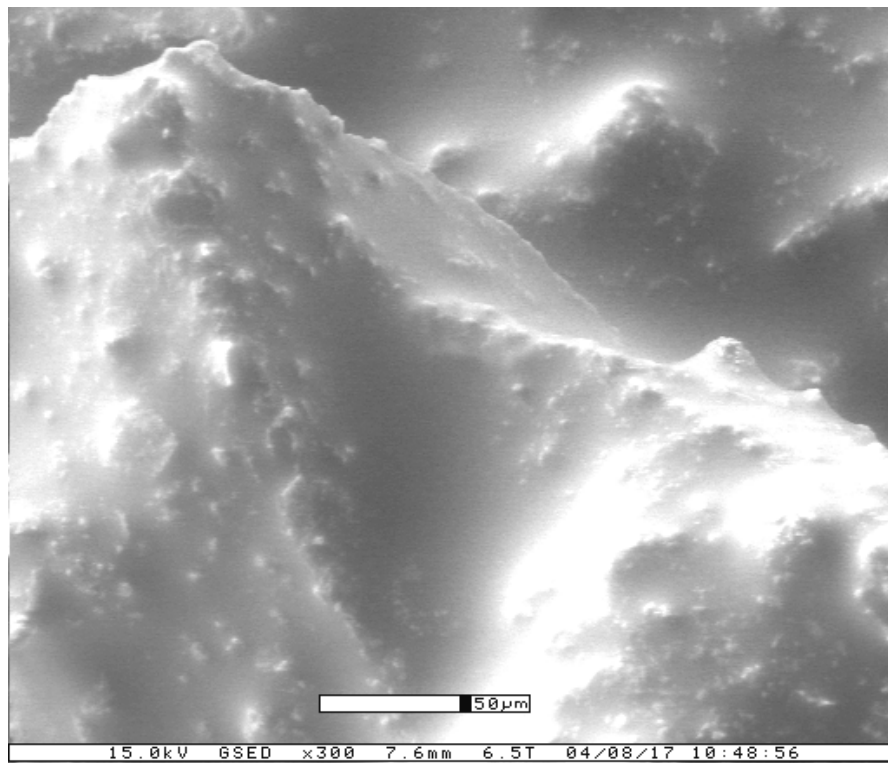


**Figure 2.2. Energy-dispersive x-ray spectroscopy (EDS) plot of control bone before conservation.** (courtesy of Microscopy and Image Center at Texas A&M University)

Bone treated with silicone oil was evaluated using SEM and ESEM. The silicone oil sample does show penetration when compared to the control (Figure 2.3). When the sample was analyzed using EDS, there was an increase in the silicon (Si) content from the control. This proves that the silicone oil penetrated into the bone. The analyses showed the same elemental composition as the control bone, but due to the penetration of the silicone oil, these elements were detected at a lower amount. A new element, vanadium (V), was detected indicating TPT Titanate catalyst was used in the silicone oil process (Figure 2.4).

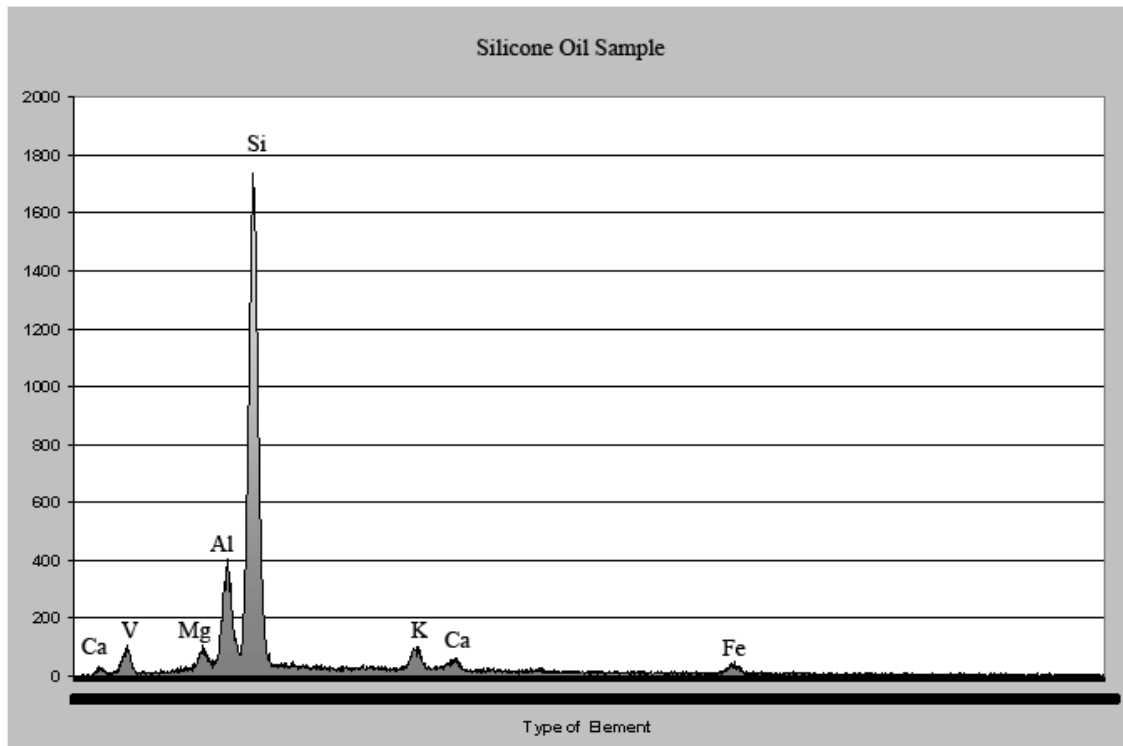


(a)



(b)

**Figure 2.3. Environmental scanning electron microscopy (ESEM). a) ESEM image of non-treated bone. b) ESEM picture of bone treated with silicone oil. (provided by Microscopy and Image Center at Texas A&M University)**

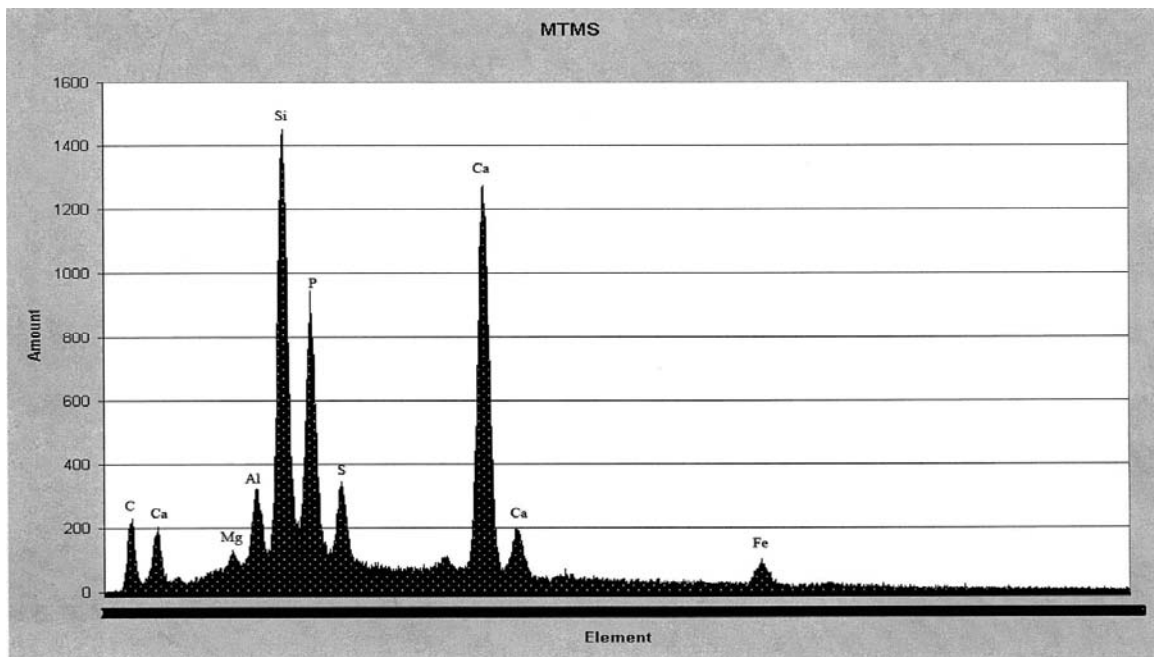


**Figure 2.4. Energy-dispersive x-ray spectroscopy (EDS) plot of silicone oil treated bone. (provided by Microscopy and Image Center at Texas A&M University)**

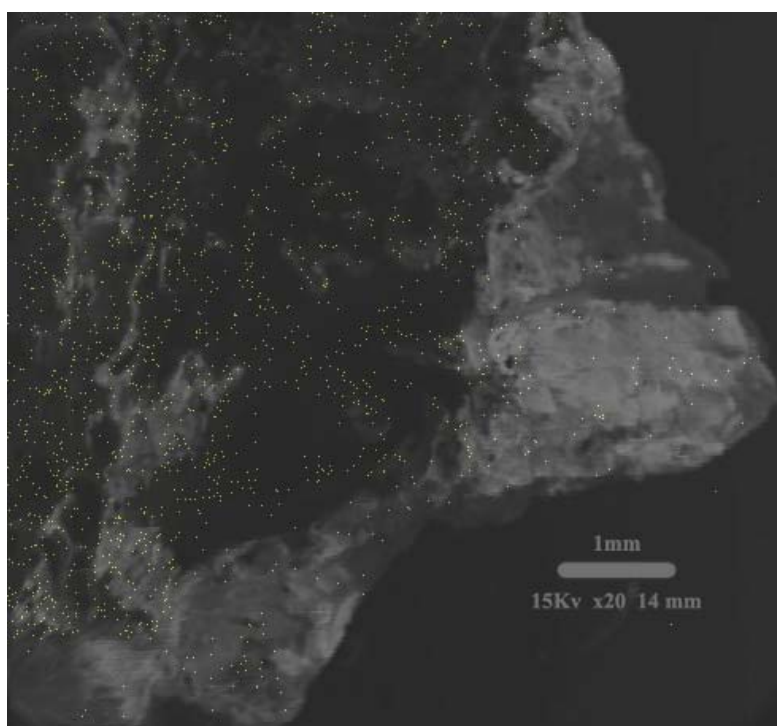
Methyltrimethoxysilane treated bone was evaluated using SEM. The bone sample treated with MTMS showed penetration when compared to the control (Figure 2.5). EDS testing detected an increase in silicon (Si) and carbon (C). Silane is a component in the chemical makeup of MTMS. The silane chemical formula is made up of silicon and hydrogen ( $\text{SiH}_4$ ). Furthermore, MTMS molecular formula,  $\text{C}_4\text{H}_{12}\text{SiO}_3$ , is made up of carbon, hydrogen, silicon, and oxygen.

Using the EDS spectrum, a number of x-ray maps were produced to illustrate penetration of silicon before and after conservation. Figure 2.6a shows the amount of

silicon (yellow dots) indicated by the EDS before conservation. Figure 2.6b shows the amount of silicon after conservation. Even though the EDS mapping does not visually demonstrate a noticeable increase in silicon, the before and after weight percentage (in red) confirms the dramatic increase. The percentage represents the amount of silicon detected when the calibration curve ratio for silicon is converted to weight percentages (Friel 2005: 28).



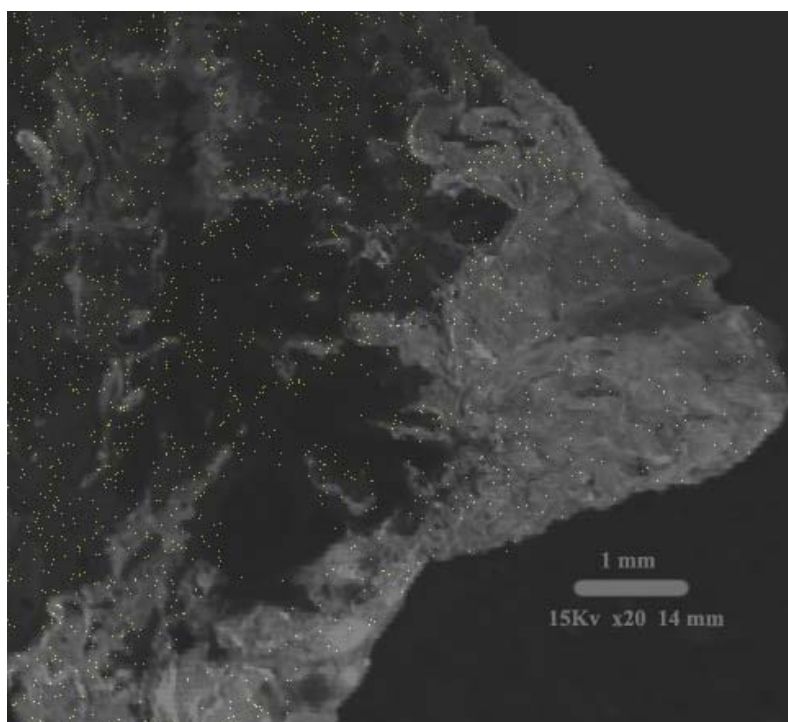
**Figure 2.5. Energy-dispersive x-ray spectroscopy (EDS) plot of methyltrimethoxysilane coated bone sample. (courtesy of Microscopy and Image Center at Texas A&M University)**



Element	Wt%	ChiSquared	Z Corr	A Corr	F Corr
Si	0.73	0.73	1.149	1.110	0.999
C	97.30	5.34	0.996	1.277	1.000
P	0.57	0.73	1.194	1.045	0.999
Fe	0.26	1.33	1.343	0.972	1.000
Ca	0.78	1.10	1.205	0.952	0.999
Mg	0.05	0.73	1.134	1.518	0.999
Al	0.31	0.73	1.175	1.253	0.998
<b>Total</b>	<b>100.00</b>	<b>2.12</b>			

(a)

**Figure 2.6. Energy-dispersive x-ray spectroscopy (EDS). a) EDS map of untreated or control bone sample. The yellow dots show the amount of silicon (Si) before conservation.**



Element	Wt%	ChiSquared	Z Corr	A Corr	F Corr
Si	20.89	1.11	0.995	1.403	0.993
C	55.67	6.24	0.859	19.745	1.000
P	5.93	1.11	1.034	1.959	0.995
Fe	1.55	2.45	1.162	1.049	1.000
Ca	9.49	0.54	1.044	1.161	0.998
Mg	0.48	1.11	0.981	1.556	0.975
Al	5.98	1.11	1.017	1.328	0.969
<b>Total</b>	<b>100.00</b>	<b>3.06</b>			

(b)

**Figure 2.6. Continue. b) Energy-dispersive x-ray spectroscopy (EDS) map of methyltrimethoxysilane treated bone sample. The yellow dots show the amount of silicon (Si) after conservation. The chart below the picture shows the weight percentage of silicon after conservation. (Courtesy of Microscopy and Image Center at Texas A&M University)**

### Experiments with MTMS Treated Bone

Further studies and experiments were conducted on methyltrimethoxysilane. These studies focused on how compatible MTMS treated bone was with other consolidants. The experiment looked at the penetration, stability, and its reaction when Rhoplex WS-24, Paleo-bond, polyvinyl acetate with a viscosity of 25, Acryloid B-72, and Starbond EM-02 were applied. For a thorough comparative analysis, the results were assembled in Tables 3 and 4.

Table 3. The before (B) and after (A) weight of conserving already MTMS treated bone.

Consolidant w/ MTMS	B. Weight	B. Length	B. Width	B. Depth	A. Weight	A. Length	A. Width	A. Depth
Rhoplex WS-24	10 grams	30.3 mm	21.6 mm	18.2 mm	10 mm	30.3 mm	21.6 mm	18.2 mm
Paleo-bond	2 grams	23.4 mm	23.6 mm	10.7 mm	1 gram	23.4 mm	23.6 mm	10.7 mm
Polyvinyl acetate V25	9 grams	58.2 mm	37.6 mm	17.8 mm	10 g	58.5	37.6	17.8
Acryloid B-72	3 grams	26 mm	20.4 mm	14.8 mm	2 g	26 mm	20.4	14.8
Starbond EM-02	17 grams	71.1 mm	39.2 mm	18.4 mm	18 g	71.1 mm	39.2	18.4



Table 4. Observing and measuring the penetration, stability, and strength of each consolidant on MTMS treated bone.

<b>Consolidant w/ MTMS</b>	<b>Pene.</b>	<b>Stability</b>	<b>Strength</b>	<b>Comments</b>
Rhoplex WS-24	0	2	2	MTMS repels water-base consolidants
Paleo-bond	3	2	2	white crystal on surface
Polyvinyl acetate with V25	3	2	2(1)	minimal to no glossy film: 1 for strength of porous area
Acryloid B-72	3	2	2(1)	no glossy film; 1for strength of porous area
Starbond EM-02	4	2	2	glossy film; may leave glue residue on surface; causes a volatile reaction to MTMS if used in excess

Stability (compare to control)

0 – Control  
 1 – Moderate flaking  
 2 – minimal to no flaking

Strength (under pressure)

0- Maximum crumbling  
 1 – moderate crumbling  
 2 – minimal or no crumbling

Penetration

0 – no penetration  
 1 – surface only  
 2 - surface/minimal  
 3 – surface/moderate  
 4 –surface/maximum

MTMS and Rhoplex WS-24

The first consolidant used on MTMS treated bone was Rhoplex WS-24, a water-based polymer. MTMS is not water-soluble consolidant, so Rhoplex WS-24 should not penetrate into the bone. After applying Rhoplex WS-24, there was no penetration. This concludes that Rhoplex WS-24 is not a consolidant compatible with MTMS.

MTMS and Paleo-bond

The next consolidant in the experiment was Paleo-bond. After this particular consolidant was applied to the MTMS treated bone, it showed Paleo-bond to permeate the surface and moderately into the bone's cancellous region. There was minimal to no

flaking, and minimal to no crumbling under pressure. There were observable crystals on the surface of the bone sample.

#### MTMS and polyvinyl acetate

Polyvinyl acetate was evaluated on MTMS treated bone. It was immersed in a solution consisting of 90% acetone and 10% by weight PVA with a viscosity of 25 for one week. After treatment, penetration was observed on the surface and moderately into the bone's cancellous region. There was minimal to no flaking observed. When adding pressure, the sample showed minimal to no crumbling on the surface, but there was moderate crumbling in the cancellous region when first taken out of the solution. This is due to the high percentage of acetone. After the acetone evaporated, the porous interior reverted back to its hardened state. There was minimal to no glossy film upon further observation. The quantitative results indicate PVA and MTMS are compatible.

#### MTMS and Acryloid B-72

Acryloid B-72 was evaluated on MTMS treated bone. It was immersed in a solution consisting of 90% acetone and 10% by weight Acryloid B-72 for one week. Just like the PVA V25 consolidant, penetration was observed on the surface and moderately into the bone's cancellous region. There was minimal to no flaking observed. When adding pressure, there was minimal or no crumbling on the surface, but there was moderate crumbling in the porous interior. After the acetone evaporated, the porous interior reverted back to its hardened state. There was an observable glossy film on the surface. Just like PVA, quantitative results show Acryloid B-72 and MTMS to be a compatible mixture.

The last consolidant was Starbond EM-02. This particular solution can be used as an adhesive and a consolidant agent at different viscosities. After applying Starbond EM-02 to the MTMS treated bone, a noticeable reaction occurred. The treated sample became hot causing it to smoke. Further research showed that MTMS did not cause this reaction. Instead, Starbond EM-02 and the experimenter caused this volatile reaction. The inadvertent application of an excessive amount of Starbond EM-02 and its catalyst caused extreme heat to radiate from the bone. After discerning this error, another experiment under controlled conditions was conducted.

The second experiment provided better results than the previous. The sample was not excessively covered with Starbond EM-02, and the catalyst was applied sparingly. This consolidant showed penetration at the surface, with maximum penetration in the interior region. There was minimal to no flaking, as well as minimal to no crumbling under pressure. Aesthetically, there was a glossy film on the surface. The quantitative observations showed Starbond EM-02 could be used as an adhesive when a proper amount is applied to the MTMS treated bone.

After looking at the results of each consolidant used in these experiments, one was chosen to help preserve the integrity of the Columbian mammoth's skeletal remains. These results will be discussed in detail in Chapter III.

## **CHAPTER III**

### **EXPERIMENTAL RESULTS AND DISCUSSION**

#### **Introduction**

Comparative experiments and analyses conducted in Chapter II showed all samples had some observable penetration and/or coating, as well as other desirable properties to conserve sub-fossil bone. However, only silicone oil, methyltrimethoxysilane (MTMS), and MTMS with a catalyst proves to be the best consolidants to stabilize and strengthen the mammoth's remains.

#### **Results and Discussion**

The silicone oil treatment consisted of using silicone oil with a cross-linker, MTMS. In the treatment process, silicone oil was impregnated into the bones' voids to prevent foreign elements from further deteriorating and weakening its structure. The final stage consisted of using a catalyst, such as TPT Titanate, to speed up and finalize the polymerization process. Qualitative analyses using ESEM and SEM illustrated that silicone oil disperses deep into the bone's structure, adding the strength needed to endure further cleaning, gluing, and handling. Another benefit of using silicone is that it does not retain a glossy film or leaves any sticky residue on the surface. In addition, it is not sensitive to temperature changes and is known for its longevity.

Silicone oil has proven to be the ideal treatment for sub-fossil and waterlogged bone. However, methyltrimethoxysilane (MTMS) was the most available and cost effective consolidant for this project. MTMS is a tight silane polymer with a low viscosity that reacts to bone's cell walls and to each other allowing a ridged bond to

occur. By immersing or topically applying 100% MTMS, it stabilizes and strengthens bone for further cleaning, gluing, or handling to occur. Exposing a catalyst to the MTMS treated bone speeds up the polymerization process to further bolster the bone's structure. The only disadvantage was the color change of the bone when using a catalyst.

MTMS had no observable discoloration and quickly bonded to the bone's cell walls. Qualitative analyses using SEM and EDS further demonstrated MTMS's penetration into the bone's structure adding support and durability. In addition, its low viscosity allowed it to impregnate easily into the bone's matrix. Another benefit of MTMS is its compatibility with other polymer-based consolidants, such as PVA or Acryloid B-72, when further conservation or gluing is needed. This is an exceptional consolidant that strengthens and stabilizes bone. Because of this consolidant's properties and availability, the mammoth's mandibles, ulna, radius, distal femur, and tooth were conserved using MTMS.

## CHAPTER IV

### AN INNOVATIVE METHOD FOR BONE CONSERVATION

#### **Introduction**

After testing and analyzing various consolidants, methyltrimethoxysilane was chosen for the preservation of the Columbian mammoth's ulna, radius, right and left mandible, distal femur, and tooth. This silane polymer allowed the bones to stabilize quickly to further facilitate cleaning and restoration. MTMS is a good consolidant for very fragile bone and sub-fossilized bone, but can be used on stable bone.

This chapter focuses on the conservation and restoration methods used on the mammoth. Even though there are guidelines relating to appropriate cleaning and preservation of an artifact, one should adapt to each artifact to ensure its integrity. Accordingly, the methods presented here are only guidelines to follow when conserving bone. During cleaning and preserving, inevitably, there will be methods which do not work. These will be mentioned to shorten the learning curve for working with fragile material. Each method is discussed in detail below starting with the frailest bone, the radius.

#### **Radius**

The radius articulates with the humerus at the elbow. Only the distal end of the radius was recovered. Once the cast was removed, noticeable new cracks and breaks were visible due to drying. The radius had an observable amount of manganese encrustation and discoloration on its surfaces (Figure 4.1).



**Figure 4.1. The radius after removing the cast.**

Due to the encrustation's reaction to the acidic tissue paper, it was easy to remove the first few layers using a wooden tongue depressor. Underlying encrustation was harder to remove, necessitating the use of stainless steel dental tools. Because the radius's stability was questionable, the decision was made to begin consolidation before extensive conservation began. Continuing to clean the bone at this time would be detrimental to its overall integrity.

MTMS was topically applied to the surface of the radius. Mask, gloves, and good ventilation was required before conserving with MTMS. Using a Nalgene Plastic Bottle with spout, MTMS was applied to the surface and to any cracks, thus speeding up the penetration rate (Figure 4.2). After seven saturated applications of MTMS, the radius was stable enough to continue cleaning.

After conserving the radius, each fragment was marked before it was transported from the APRL lab to the Center for the Study of the First American Lab (CSFA) at Texas A&M University directed by Dr. Mike Waters (Figure 4.3). Next, it was tightly wrapped in plastic wrap to hold the pieces together for the quick transfer from the countertop onto a sand-filled cart. With the help of other graduate students, it was transported to the CSFA Lab for further cleaning and reconstruction.



**Figure 4.2. Showing the methodology of conserving the Columbian mammoth using methyltrimethoxysilane (photography by Robert Larsen).**





**Figure 4.3. Marking the radius before taking it apart piece by piece.**

The cleaning process began by removing each individually marked fragment and placing it in its correct orientation on a covered tray (Figure 4.4). The MTMS bonded with the water in the soil and encrustation near the surface and inside the radius making cleaning difficult. Therefore, a more abrasive tool, an air scribe, was utilized to mechanically remove the encrustation (Figure 4.5). Some of the encrustation was left embedded in the radius because it would cause further damage if removed (Figure 4.6).



**Figure 4.4. Removing the pieces from the radius for cleaning and reconstruction.**



**Figure 4.5.** Air scribing the distal end of the radius (photography by Joshua Daniel).



**Figure 4.6.** The results after air scribing the distal end of the radius.

The reconstruction process began by gluing fragments together using glue produced at the Conservation Research Lab (CRL) at Texas A&M University. The adhesive's solution is 50% Acryloid B-72 and 50% acetone by weight mixed with cabosil (fine ground glass). It was excellent for gluing small, medium and most large pieces together. Due to the amount of solvent in the adhesive, each glued section needed at least 24 hours to set. Due to the mass and type of longitudinal breaks in the bone, stabilization was difficult when using Acryloid B-72 adhesive. Therefore, a cyanoacrylate called Starbond EM-02 was chosen to stabilize the larger bone fragments. The only set back was that it adheres to the bone's surface quickly and is hard to remove once its set. Also, if used in excess, it will turn an area of the bone a greenish tint (see Chapter II, Figure 2.1).

While gluing the radius, many of the fragments did not align properly due to previous shrinking and warping resulting in obvious gaps and cracks. The experimental test on MTMS in Chapter II does not show any shrinkage or warping. Figure 4.7a shows the radius without observable crack while in the field, while figure 4.7b reveals the radius with observable shrinkage and cracks. Once bone has been allowed to dry at an uncontrolled rate, it will crack and warp. The ideal procedure is to bring the high relative humidity (RH) of any wet artifact slowly down until it matches the storage area's relative humidity (McCarty 2002: 13). This statement along with observable evidence demonstrates that the warping and shrinkage was caused by the drying process before conservation ever took place.



(a)



(b)

**Figure 4.7. Radius. a) The radius's appearance at the Vernor Site. b) Observable cracks and breaks on the radius due to the fluctuation in temperature during the drying out process.**

The gaps, cracks, and missing sections were filled with Paleo-bond epoxy putty to achieve a more pleasing specimen and further stabilize the radius. Paleo-bond epoxy putty is very pliable, durable and molds to any surface. Furthermore, it takes 24 hours to cure giving ample time for the conservator to work.

The distal section of the radius was a challenge to repair. There was evidence of crumbling where encrustation was once located possibly due the inability of the MTMS to penetrate into these areas. So, the next step included immersing the distal end in a solution of 80% Acetone and 20% Acryloid B-72. When the radius was immersed, off-gassing was observed in the form of bubbles, showing there was displacement of air with the solution. The bone was left in the solution for 24 hours. After the allocated time, it was removed and left to dry. The radius's distal end was stable, and no further crumbling was observed. The downside was the glossy film on the surface from the Acryloid B-72. Using Paleo-bond epoxy putty, cracks and breaks were repaired. The proximal and distal ends were not constructed together due to time constraints. The end result was a stable radius for future handling and exhibits (Figure 4.8 a-d).



(a)



(b)



(c)

**Figure 4.8. Conserved radius. a) The finished product of the radius's distal end. b) The distal end of the radius after conserving. c) The distal end of the radius.**



(d)

**Figure 4.8. Continue. d) The distal end of the radius and the shaft.**

### **Left Mandible**

The mandible is the jaw bone attached to the lower section of the cranium on a Columbian mammoth. The top section of the left mandible was in pieces when the cast was removed (Figure 4.9 a and b). The cancellous or porous tissue was very friable and crumbled easily. On the other hand, the outer bone was stable enough to begin cleaning. Most of the encrustation and soil deposits were removed by wooden tongue depressors or dental tools before relocating it to the CSFA lab.

Once at the CSFA lab, the cleaning process continued with the inside of the lower portion of the mandible. The soil was holding it in place, so each individual broken or cracked piece was marked (Figure 4.10) and placed on a covered tray (Figure 4.11 a and b).





(a)



(b)

**Figure 4.9.** Left mandible. a) The side view of the left top portion of mandible's break. b) The back view of the top portion of the mandible's break.



**Figure 4.10.** Marking the top portion of the left mandible before disassembling the pieces.



**Figure 4.11. The first stages of cleaning after removing the marked pieces from the top portion of the left mandible.**

Next, the left mandible's bottom portion, which included the molar, was cleaned using dental tools. The encrustation was difficult to remove, so a diluted solution of 10% sodium hexametaphosphate to 90% water was applied. This solution loosened the encrustation for easier removal. New cracks and breaks were observed while cleaning requiring the mandible to be conserved using 100% MTMS.

The top portion of the left mandible's marked bone fragments were immersed in MTMS. The immersion time varied due to the each fragment's size and porosity. On average, the smaller pieces were immersed for 30 minutes, while the larger pieces were immersed for 1 hour. Another way to monitor the penetration rate of MTMS is to observe the off-gassing process. When the replacement of air with MTMS is complete, there should be no more off-gassing or observable bubbling. The bottom portion of the

mandible was conserved by topically applying MTMS. After ten applications of MTMS, the left mandible and molar were stable enough to continue cleaning.

The reconstruction was difficult due to the irregular surface of each broken fragment. Each marked piece was glued using Acryloid B-72 and allowed to dry for 24 hours. Sand-filled bags were strategically placed for stability during the 24 hour period. There were still some pieces that did not properly fit back together. There are two reasons for the misalignment. Although the break's surface may look clean and smooth, microscopically it is very irregular and difficult to reassemble. The other reason is when objects break, the "newly fracture surface becomes contaminated by oxygen, water, or other chemicals in the environment" making the surface over a short period more irregular and friable (Wheatcroft 1994: 13-14). These breaks and gaps were filled in with epoxy putty to achieve a pleasing exterior look, as well as add structural support.

The next step involved reinforcing the top portion to the lower portion of the mandible (Figure 4.12). Dr. Helen Dewolf, Head Conservator at the Conservation Research Lab (CRL) at Texas A&M University, suggested using a transparent woven material with Hysol™ Resin Epoxy RE2039 with 30% by weight Hysol™ Hardener HD 3561. This mold would give extra support to the mandible's top portion.



**Figure 4.12. The left mandible after gluing but before adding a mold to help stabilize the upper jaw. The arrow represents where the bottom and top portion of the mandible are attached.**

Three long strips of Paleo-bond epoxy putty were placed along the mandible's interior. While the epoxy putty was pliable, small pieces of steel wire were placed upright in the epoxy. Thus, allowing the support mold to have a mechanism to attach to instead of the bone itself (Figure 4.13 a-c).

**(a)****(b)****(c)**

**Figure 4.13. Left mandible reconstruction. a) Bottom section of the mandible with the epoxy putty and wire. b) Overview of the inside of the mandible. c) Side view of the mandible with the epoxy putty and steel wire.**

With the help of Michael West, former Texas A&M University nautical graduate student, the support mold was constructed. Small woven strips covered with Hysol™ Resin Epoxy RE2039 with 30% by weight Hysol™ Hardener HD 3561 were placed along the left mandible's interior. Due to the slippery nature of the epoxy resin, the woven material would not adhere to the wire or epoxy putty long enough to set. Subsequently, the plan to use this particular support mold was not achieved.

A decision was made to remove the steel wires and add another layer of Paleobond epoxy putty giving the strength needed without the use of a support mold. The finished product was a stable and reconstructed left mandible (Figure 4.14 a-c).



(a)

**Figure 4.14. Conserved left mandible. a) The overview of the left mandible after conservation.**



(b)



(c)

**Figure 4.14. Continue. b) The exterior side of the left mandible after conservation. c) The interior side of the left mandible after conservation. (photography by Charlotte Pevny)**

### **Right Mandible**

The right mandible was cast by itself, so it was stable enough to remove most of the soil before conserving. There were observable cracks and breaks, as well as discoloration on the surface. Just like the left mandible, it was placed on a sand-filled cart and transported to CSFA to resume cleaning.

Cleaning took some time since there was a massive amount of manganese encrustation and tissue paper adhering to its surface (Figure 4.15). By using wooden tongue depressors, dental tools, and soft bristle toothbrushes, the majority of the tissue paper and encrustation was removed. Due to environmental fluctuations, new cracks began to occur requiring the mandible to be consolidated with 100% MTMS.



**Figure 4.15. The right mandible's appearance while in the process of cleaning.**

The conservation methodology for the right mandible was the same as the left mandible. MTMS was topically applied to the surface. After ten applications, the right mandible was stable enough for reconstruction.

The reconstruction of the right mandible was not complicated in comparison to the left mandible. By using Acryloid B-72 glue, pieces were assembled back to their respective places. The breaks and gaps were filled-in using Paleo-bond epoxy putty making a stable and aesthetically pleasing right mandible (Figure 4.16 a-c).





(a)



(b)

**Figure 4.16. Conserved right mandible. a) The overview of the right mandible after conservation. b) The interior view of the right mandible after conservation. (photography by Charlotte Pevny)**



(c)

**Figure 4.16. Continue. c) The exterior of the right mandible after conservation.**  
(photography by Charlotte Pevny)

## **Ulna**

The ulna lies directly behind the radius and connects to the humerus. Only the proximal end and part of the shaft was recovered. Once the cast was removed, there were no observable cracks, but the bone suffered a noticeable amount of discoloration. (Figure 4.17).



**Figure 4.17. Photograph of the discoloration caused by acidic tissue paper and aluminum foil.**

Soil, tissue paper, and encrustation were removed using tongue depressors, a soft bristle toothbrush, and a water pick. The application of water caused an adverse effect resulting in the proximal end to break into three fragments. The ideal cleaning method is to remove surface dirt from stable bone by using water (Hamilton 2000: 1). But in this case, the bone was not as structurally sound as previously thought. Fortunately, these breaks were repaired.

Dental tools and an air scribe were used to remove the underlying encrustation. Some of the soil and encrustation was embedded deep into the bone's surface and removal was not possible. After cleaning, there was still noticeable surface discoloration and encrustation on the ulna (Figure 4.18).



**Figure 4.18. After cleaning the ulna, there was still noticeable discoloration and encrustation on the surface.**

Before repairing could begin, methyltrimethoxysilane was topically applied to the shaft and the three broken fragments (see Figure 4.2). After ten applications of

MTMS, the ulna was stable enough to continue cleaning and begin reconstruction.

Using Acryloid B-72 glue, each piece was assembled in its appropriate place.

Afterwards, gaps and cracks were filled in using Paleo-bond epoxy putty producing a more aesthetically pleasing ulna (Figure 4.19 a and b).



(a)



(b)

**Figure 4.19. Conserved ulna. a) The proximal end of the ulna after conservation. b) View of the ulna's other side after conservation. (photography by Charlotte Pevny)**

### **Distal Femur**

The femur is the largest bones in the rear legs of a Columbian mammoth. Only the distal end of the femur was recovered. Due to its small mass, it was not cast but wrapped in wet tissue paper. It was placed in an oversized plastic bag for transportation to Texas A&M University. Once the tissue paper was removed, mold was detected. The femur's surface was very water-worn with little of the cortical bone left, causing most of the cancellous tissue or porous area to be exposed (Horie 1982: 1-2) (Figure 4.20).



**Figure 4.20. Photography of distal femur before conservation.**

With the cancellous tissue exposed, mechanically cleaning was difficult. The only option left was to use water. Using a water pick and a soft bristle toothbrush, the soil was removed from various areas. Unfortunately, the water pick was not abrasive enough to remove the soil. As a result, it was immersed in a solution of 10% sodium

hexametaphosphate with 90% water to loosen the soil. After taking it out of the solution, it was immediately dried. At this point, no new observable cracks were found and the soil was removed using a long thin wooden pick in conjunction with a water pick.

Conserving the distal femur was manageable due to its size. It was immersed in 100% solution of methyltrimethoxysilane for two hours to ensure complete penetration. Unlike the left mandible's fragments, the immersion time was extended by 30 minutes due to the femur's volume. The results showed no further crumbling, and the femur was stable enough to handle (Figure 4.21 a and b).



(a)

**Figure 4.21. Conserved distal femur. a) The distal femur after conservation.**



(b)

**Figure 4.21. Continue. b) Another view of the distal femur after conservation.**

### **Tooth**

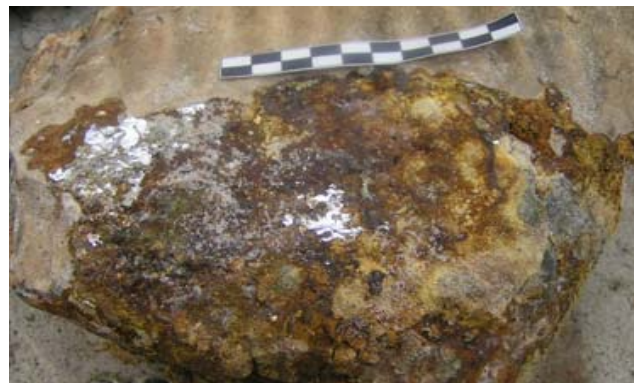
Mammoth teeth are significant because they help distinguish between species, as well as determines its' approximate age. Both the upper and lower teeth have a tight cellular structure and multiple layers of enamel to help retain its structural integrity (Haynes 1991: 6-7). This specific tooth was not cast due to its small size. As an alternative, it was wrapped in wet tissue paper. Foil was added on top of the wet tissue paper following a layer of bubble wrap as a protector. It was transported to Texas A&M University and was not examined until later that year.

The tooth was stable but the surface was discolored and flaking (Figure 4.22 a-c). The tissue paper and foil were easily removed using wooden tongue depressors and dental tools. The manganese encrustation was embedded on the tooth's surface requiring utilization of a more abrasive tool, an air scribe. By applying a small amount

of pressure with the air scribe, the encrustation was easily removed. The surface discoloration could not be removed.



(a)



(b)

**Figure 4.22. Tooth. a) Showing the discoloration on one side of the tooth. b) A closer view of the discoloration with pieces of foil adhering to it.**





(c)

**Figure 4.22. Continue. c) The top view of the tooth before cleaning.**

Because teeth have a different organic structure, a different approach to consolidating the tooth was taken. It was immersed in a 100% solution of MTMS. Methyltrimethoxysilane is capable of penetrating deep within tightly laced organic or inorganic structures because of its low viscosity (Charola et. al 1984: 177). After two hours, there was still noticeable off-gassing or bubbling occurring; therefore it stayed in MTMS for an additional hour. The tooth's results were exceptional. It showed stability and no surface flaking after conservation (Figure 4.23 a-c). No reconstruction was necessary with the tooth.



(a)



(b)

**Figure 4.23. Conserved tooth. a) The final results of the tooth after conservation. b) Other side of the tooth after conservation.**



(c)

**Figure 4.23. Continue. c) The top view of the tooth after conservation.**

The overall results of using 100% methyltrimethoxysilane on the mammoth's remains were quite exceptionally. MTMS has provided the strength, stability, and natural appearance needed for future examinations and viewing to occur.

## CHAPTER V

### CONCLUSION

#### **Summary and Conclusion**

It is astonishing to imagine that 66,000 years ago the modern coastal region of Texas was abundant with 13 foot tall Columbian mammoths. One particular Columbian mammoth did not leave the coastal region, but died in the sandy soils of Texas. In January and February of 2004, faculty and students from the Center for the Study of First Americans at Texas A&M University, in conjunction with the Brazosport Archaeological Society uncovered a disarticulated Columbian mammoth in Clute, Texas. Two tusks, a left and right mandible, distal femur, ulna, radius, and a tooth were recovered.

This specific Columbian mammoth is one of the few mammoths discovered along the Texas coast, and conserving it was of the utmost importance for future studies in the Pleistocene Epoch. A literary review was conducted to study both the historical and modern perspectives on bone conservation to help answer the question of what is the best consolidant for stabilizing the mammoth's skeletal remains. After reviewing different perspectives on conserving bone, a comparative analysis was conducted using both polymer-base consolidants and cyanoacrylates.

Experiments were conducted on various bone fragments to compare the consolidants' properties of conservation materials. The following consolidants were examined and tested: silicone oil, polyvinyl acetate (PVA) with a viscosity of 25, Acryloid B-72, Butvar 98, Starbond EM-02, methyltrimethoxysilane (MTMS), MTMS

with a catalyst, Paleo-bond, and Rhoplex (Primal) WS-24. Penetration, strength, stability, and appearance of each bone sample were evaluated in the research. Scanning Electron Microscopy (SEM) and Environmental Scanning Electron Microscopy (ESEM) were utilized to map penetration depth of silicone oil and methyltrimethoxysilane.

Final analyses showed that all samples had received some observable penetration and/or coating. However, silicone oil technology, methyltrimethoxysilane (MTMS), and MTMS with a catalyst showed the best stability and penetration on sub-fossilized bone. The silicone oil treatment consisted of using silicone oil with a cross-linker, MTMS. In this treatment process, silicone oil was immersed into the bones' cancellous framework to prevent outside elements from further deteriorating and weakening its structure. The final stage consists of using of a catalyst, such as TPT Titanate, to finalize or speed up the polymerization process. Silicone oil does not retain a glossy film, nor does it leave any sticky residue on the surface. Another benefit of this treatment is that it provides strength needed to endure further cleaning, gluing, and handling.

Quantitative and qualitative experiments have showed that silicone oil technology may be the ideal treatment for sub-fossilized and waterlogged bone. However, methyltrimethoxysilane (MTMS) was the most available and cost effective consolidant for this project. Methyltrimethoxysilane (MTMS) is a tight, ridged silane polymer with a low viscosity. By immersing or topically applying 100% MTMS, the bones' structure is strengthened. Exposing a catalyst to the MTMS treated bone speeds up the polymerization process to further stabilize the bone's structure. The only drawback was the color change of the bone when using a catalyst.

MTMS has no observable discoloration and its low viscosity allowed it to impregnate into and around the bones' cancellous tissue to strengthen and stabilize its structure. MTMS is compatible with other polymer-based consolidants when further conservation or gluing is needed. This is an exceptional consolidant that strengthens bone, thus preserving its integrity.

Further studies are needed on the polymer-based consolidant MTMS to determine its mechanism and long-term degree of polymerization, evaluate long-term environmental factors, and conduct test on non-sub-fossilized bone from both dry and wet sites. This research helped to open new avenues to different methods in preserving sub-fossilized bone, as well as broaden our understanding of sub-fossilized bone conservation. Hopefully, this will not be the end, but the beginning of an in-depth study on new and innovative methods for conserving bone.

**REFERENCES CITED**

- Agenbroad, L.D.  
1999 *Columbian Mammoth (Mammuthus columbi)*. Mammoth Site Research Notes No. 1. Mammoth Site of Hot Springs. Hot Springs, South Dakota.
- Bronstein, Allen  
1981 The Chemistry of Polyethylene Glycol. Proceedings of the ICOM Waterlogged Wood Working Group Conference. Ottawa, Canada p.279-285.
- Charola, A.E., G.E. Wheeler, and G.G. Freund  
1984 The Influence of Relative Humidity in the Polymerization of Methyltrimethoxysilane. *Preprints of the Contributions to the Paris Congress, 2-8 September Adhesives and Consolidants* 177-181. London.
- City of Clute  
2005 *Consumer Confidence Report 2005: Drinking Water Quality Report*. City of Clute and U.S. Environmental Protection Agency. Copies available from Clute City Hall, Clute, Texas.
- Cronyn, J.M.  
1990 *The Elements of Archaeological Conservation*. Routledge: London and New York.
- Davis, Simon J.M.  
1987 *The Archaeology of Animals*. Yale University Press: New Haven, Connecticut.
- Dewolf, Helen  
2005 Personal Communication. Texas A&M University. College Station, Texas
- Down, Jane L. and Elzbieta Kaminska  
2006 A Preliminary Study of the Degradation of Cyanoacrylate Adhesives in the Presences and Absence of Fossil Material. *Journal of Vertebrate Paleontology*, 26(3): 519-525.
- Elder, A., S. Madsen, G. Brown, C. Herbel, C. Collins, et al.  
1997 Adhesives and consolidants in Geological and Paleontolical Conservation: A Wall Chart. SPNHC Leaflets, Volume 1, Number 2, Spring.

Friel, John J.

2005 *X-Ray and Image Analysis in Electron Microscopy 2<sup>nd</sup> Edition*. Princeton Gamma-Tech Inc. Rocky Hill, New Jersey.

Glover, Roy, with Robert W. Henry, and Ronald S. Wade

2003 Passivation Polymer Technology: A Next Generation Process for Biological Specimen Preservation. Electronic Document, <http://nautrach.tamu.edu/APRL/PRESS01.htm> (accessed November 11, 2003).

Hamilton, Donny L.

2000 Conservation of Bone, Ivory, Teeth and Antler. Conservation Research Laboratory, Texas A&M University. Electronic document. <http://nautarch.tamu.edu/class/anth605/File3.htm> (accessed January 26, 2004).

Haynes, Gary

1991 *Mammoths, Mastodons, and Elephants : Biology, Behavior, and the Fossil Record*. Cambridge University Press: Cambridge: New York.

Hedges, R.E.M.

1987 Potential Information from Archaeological Bone: It's Recovery and Preservation. In *Archaeological Bone, Antler, Horn*. edited by Starling, K & Watkinson, D. UKIC Occasional Papers, No. 5. London: UKIC 22-23.

Horie, C.V.

1982 Reversibility of Polymer Treatment, *Resins in Conservation*, Proceeding of the Symposium, Edinburgh 1982, edited by J.O. Tate, N.H. Tennent and J.H. Townsend, Scottish Society for Conservation and Restoration, pp. 3-1 to 3-6.

Johnson, Jessica S.

1994 Consolidation of Archaeological Bone: A Conservation Perspective. *Journal of Field Archaeology*, 21: 221-233.

Klosowski, Jerome, with Charles Wayne Smith and Donny Leon Hamilton

2004 Conservation of organic and inorganic materials. Electronic document. <http://patft.uspto.gov> (accessed June 10, 2004).

Lister, Adrian and Paul Bahn

1994 *Mammoths*. Prentice Hall Macmillan Company: New York, New York.



McCarty, Russ

- 2002 Fossil Preparation and Conservation. Electronic Document.  
<http://www.flmnh.ufl.edu/natsci/vertpaleo/resources/prep.htm> (accessed November 11, 2002).

O'Connor, Sonia.

- 1987 The Identification of Osseous and Keratinaceous Material at York.  
In *Archaeological Bone, Antler, Horn*, edited by Starling, K. &  
Watkinson, D. UKIC Occasional Papers, No. 5. London: UKIC, 9-21.

Potapova, Olga

- 2005 Personal communication. Hot Springs Mammoth Site, Inc. Hot Springs  
South Dakota.

Rainey, Robert

- 2004 Personal Communication. Vertebrate Paleontology Lab. University of  
Texas. Austin, Texas.

Schiffer, Michael B

- 1987 Environmental Processes: The Artifact. *Formation Processes of the  
Archaeological Record*. New Mexico: University of New Mexico Press,  
Albuquerque.

Sease, Catherine

- 2003 *A Conservation Manual for the Field Archaeologist 2<sup>nd</sup> Edition*. Los  
Angeles: University of California Press.

Shelton, Sally Y. and Jessica S. Johnson

- 1995 The conservation of sub-fossil bone. In *The Care and Conservation of  
Palaeontological Material*, edited by Chris Collins, pp. 59-71.  
Butterworth-Heinemann, Ltd.: Jordan Hill, Oxford, United Kingdom.

Singley, Katherine

- 1988 *The Conservation of Archaeological Artifacts From Freshwater  
Environments*. Lake Michigan Maritime Museum. South Haven,  
Michigan

Smith, C. Wayne

- 2003 *Archaeological Conservation using Polymers*. Texas A&M University  
Press. College Station, Texas.

- 2006 Personal Communication. Texas A&M University. College Station, Texas.

- Stone, Tammy T., with David N. Dickel, and Glen H. Doran  
1990 The Preservation and Conservation of Waterlogged Bone from the  
Windover Site, Florida: A Comparison of Methods. *Journal of Field  
Field Archaeology* 17(2):177-176.
- Storch, Paul  
2003 *Field and Laboratory Methods for Handling Osseous Materials*.  
Conservation Notes No. 6. Austin: Texas Memorial Museum.
- Wheatcroft, Andrew (editor)  
1994 *Science for Conservators, Volume 3. Adhesives and Coating*.  
The Conservation Unit. Routledge, London.

## VITA

**Shanna LaRea Daniel**  
**3301 Providence**  
**Bryan, Texas 77803**  
**(979) 492-6772**

### EDUCATION

- High School Diploma, Spring Hill ISD in Longview, Texas, May 1998
- Bachelor of Liberal Arts, Stephen F. Austin State University in Nacogdoches, Texas, December 2002
- Master of Arts in Anthropology, Texas A&M University in College Station, Texas, Spring 2007

### WORK HISTORY

*2005-Present, Lab Assistant, Texas A&M University Conservation Research Lab, (979)862-7791*

I assist with the curation of maritime and terrestrial archaeological artifacts using various conservation methods (silicone oil, etc...) under the supervision of Dr. Helen Dewolf and Mr. Jim Jobling. My duties include: cleaning, conserving, and reconstructing artifacts, as well as database entry using FoxPro.

*2001-Present, Assistant Archaeologist, Victor Galan – Archaeologist, (936) 560-4670*

I assist Victor Galan with various Cultural Research Management surveys in East and West Texas, as well as processing artifacts for curation.

*2001-2003, Lab Assistant, Stephen F. Austin State University Anthropology Lab, (936)468-2457*

I was a lab assistant in the Anthropology Department under the supervision of the late Dr. Jim Corbin. My duties included the analysis and curation of artifacts from archaeology sites, cataloging the artifacts, and conserving metal nails.

*2001-2003, Museum Assistant, Stone Fort Museum, (936)468-2408*

I was a museum assistant at the Stone Fort Museum under the supervision of Mrs. Carolyn Spears, MA. My duties included properly storing museum artifacts, help display artifacts, assisting with group tours, and helping with various office and computer work such as filing, database, and answering the phone.

### EXPERIENCE & ACTIVITIES

- Thesis project: conservation of a Columbian mammoth's bones
- Historical Preservation Certificate (May 2007)
- Scanning Electron Microscopy and Environmental Scanning Electron Microscopy certified
- Conservation Certificate (May 2007)
- Internship at the Star of the Republic of Texas Museum