AN ASSESSMENT OF WATERLOGGED WOOD CONSERVATION
TECHNIQUES FOR LITTLE SALT SPRING (8SO18):
AN ARCHAIC MORTUARY POND

by

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AN ASSESSMENT OF WATERLOGGED WOOD CONSERVATION TECHNIQUES FOR LITTLE SALT SPRING (8SO18): AN ARCHAIC MORTUARY POND

Christine Anastasia Mavrick

Waterlogged wooden artifacts and ecofacts recovered at this unique Archaic site present researchers with a challenge to meet specific conservation needs. This thesis sought to address these needs through research and redundant experimentation. In over twenty experiments, commonly practiced conservation techniques were tested in an attempt to discover an easily replicable conservation method that best fit these objects. Additionally, a brand new method of waterlogged wood conservation, recently developed in Japan and employing hydrolyzed feather keratin, was tested for use on artifacts from Little Salt Spring.
CHAPTER I

INTRODUCTION

In the early 1950s, avocational SCUBA divers noted the presence of extensive bone scatters within the basin of a sinkhole while searching for new dive sites in Sarasota County, Florida (Wentz and Gifford 2007:330). It was not until 1959 that local diver and amateur archaeologist William Royal investigated this site. His initial observations and subsequent report of human skeletal material along the basin and various ledges led to further investigations. Along with Eugenie Clark, a marine biologist, Royal made a number of dives at Little Salt Spring and recovered hundreds of disarticulated human bones. While there were no complete skeletons initially found within the recesses of the sink, the skeletal material collected by Royal and Clark were sufficient to represent at least 50 individuals.

The early 1970s saw an expansion of the site investigations when the Division of Archives, History and Records Development developed a cooperative research program with the General Development Corporation, a Floridian land development company, that involved a multi-disciplinary agenda employing archaeologists, limnologists, geologists and physical anthropologists. The initial phases of research (I and II) were designed to familiarize researchers with the site through environmental evaluations and prepare for Phase III, which would involve major archaeological investigation (Florida Department of State, Division of Historical Resources [FDoS, DHR] 1972).
Today, the research complex at Little Salt Spring is outlined by a 110-acre area that was donated to the University of Miami in 1982 and also extends beyond this property to an adjacent 5-acre parcel of land owned by Sarasota County. This complex includes several important archaeological components and at least two occupations. The spring basin has yielded primarily Paleo-Indian artifacts, but also contains a Middle Archaic component associated with a Middle Archaic cemetery, which surrounds the basin and extends up into the Little Salt Slough. In addition to the spring and the slough, there is an associated Middle Archaic habitation adjacent to the cemetery (Koski et al. 2006:22).

Both the spring and the slough contain significant human skeletal remains; researchers estimate that between 100 and 1,000 burials rest in the peaty sediments of the slough. Wentz and Gifford (2007) liken Little Salt Spring to wet cemeteries or mortuary ponds such as those found at Windover in Brevard County (8BR246), Republic Groves in Hardee County (8HR4) and Bay West in Collier County (8CR200) (Figure 1). Mortuary ponds of this type are unique within the Archaic to Florida and the similarities between these sites suggest a cultural continuity throughout the region (Wentz and Gifford, 2007:334). In addition to elucidating a possible common cultural complex, Little Salt Spring has produced artifacts that date amongst the earliest recovered anywhere in the United States.

Over the past few decades, researchers at Little Salt Spring have worked diligently to unearth clues about Florida’s prehistory and in the process have recovered artifacts not only consisting of human skeletal materials, but also of animal bone, stone and wood. Preservation of these cultural items is promoted through a virtual absence of
Figure 1. Map of the state of Florida - archaeological sites containing Archaic mortuary ponds.
dissolved oxygen, a fairly constant water temperature and high numbers of dissolved minerals within the spring’s waters (Koski et al 2006:33). Artifacts recovered from the underwater basin and ledges of Little Salt Spring provide researchers with documentation of both the environment and cultural activities of some of the earliest peoples in the United States (Koski et al 2006:33). It is important, therefore, that objects removed from the spring retain as much information as possible. Waterlogged material removed from its wet environment can lose shape, size or surface detail. When removed from its environment, untreated waterlogged wood will disintegrate to almost nothing because of cellular degradation, if its water content is high enough. Therefore, once artifacts are removed from this anoxic environment, researchers are faced with the problem of developing a proper method of treatment. To date, no completely suitable method has been determined for the treatment of organic artifacts, specifically wooden artifacts, from Little Salt Spring. Very few experiments have been conducted to treat prehistoric waterlogged wood anywhere, and those that have were largely unsuccessful. In 1959, Robert M. Organ published his efforts to treat recovered wooden tools from Southern Rhodesia dating from the Lower Paleolithic. Twenty-six objects from approximately 53,000 years b.p. were treated using various methods, including freeze-drying without a pre-treatment, freeze-drying after dehydration using trimethyl carbinol and impregnation with high molecular weight polyethylene glycol employing a method of evaporation. The outcome of these experiments went very far to rule out a couple of these methods but was unable to produce results that could suggest a positive treatment for future conservators (Organ 1959:96-105). Wallace R. Ambrose included some notes on work with 10,000-year-old wooden boomerangs recovered in Australia when he reported on his
experiments with freeze-drying 2000 year old swamp wood in 1975 (Ambrose 1975:75/8/4-1 – 75/8/4-12). In the United States, Glen H. Doran and David N. Dickel reported unsatisfactory results in conserving 8000-year-old wooden stakes recovered in the 1980s from the Windover Site in Florida (Doran 2002). To date, there have been no successfully repeated conservation treatments of waterlogged prehistoric wood. This thesis seeks to provide a solution to this problem through the synthesis and evaluation of both practiced and nascent conservation methods and redundant experimentation.
CHAPTER II
SITE HISTORY

The state of Florida sits on the Florida Plateau, a geological feature that was formed over 530 million years ago during the Ordovician through volcanic activity and marine sedimentation (Allen and Main 2005:1). Since the Cretaceous, the Florida Plateau has accumulated thousands of feet of limestones and dolomites (Beck 1986:5). These limestones make up the over 100,000 square miles of the Florida Aquifer System, which extends beneath Florida, as well as parts of Alabama, Georgia and South Carolina (Figure 2). The Florida Aquifer is one of the highest producing aquifer systems in the world, according to the Florida Department of Environmental Protection (Florida Department of State, Department of Environmental Protection 2007). Over much of the central and northern parts of Florida, clastic sediments of the Miocene Hawthorn Group overlie and confine the limestones of the Florida Aquifer, however, the area along the crest and the western flank of the Ocala Uplift (Beck 1986:7) has been stripped of this clast cover through erosion, leaving exposed limestone surfaces. Within the Gulf coastal lowlands, scattered cenotes, sinkholes, lakes and springs mark low-level karst plains.

Karst landforms, such as cenotes or sinkholes, are formed through the dissolution of solid bedrock, such as limestone. Carbonic acid is formed when water, as rain, passes through the atmosphere and picks up carbon dioxide. It then passes through the soil, picking up more carbon dioxide before meeting the groundstone. Over time, acidic water
Figure 2. Florida aquifer.
creates these new landforms (Faught and Carter 1998:168). Many Paloeindian and Early Archaic sites were located near these karst features as they provided access to potable water and fauna as well as chert and wood for tool-making and building (Faught and Carter 1998:173). The central Gulf coastal region of Florida exhibits several of these cenotes and dolines that also contain valuable archaeological preserves.

Little Salt Spring is a cover-collapse, karst-solution sinkhole, or cenote, located in North Port, Florida, less than one hundred miles south of Tampa in Sarasota County. Spanning just 78 meters in diameter, the sinkhole exhibits a basin-like depression approximately 12 meters deep. At the center of the basin, a circular opening about 30 meters across opens up into a 60-meter deep sinkhole (Alvarez Zarikian et al. 2005:135; Clausen et al. 1979:610; FDoS, DHR 1972; Koski et al. 2006: 7).

Little Salt Spring was formed over 12,000 years ago when the ceiling of the solution pocket, or underground cave, collapsed and fell 60 meters to the floor of the cave (Alvarez Zarikian et al. 2005:142; Koski et al. 2006:8). The cave filled with water at a rate of approximately 0.7 centimeters a year, eventually surpassing the orifice near 8,500 b.p. and filling the surrounding basin and bringing the water level to what can be seen today on the modern landscape (Clausen et al. 1979:610).

Avocational divers first discovered the site in the early 1950s and it was later investigated by Colonel William Royal of Venice, Florida. Royal reported the discovery of significant human remains and, in fact, recovered enough skeletal material to account for at least 50 individuals along the lower slope of the basin (FDoS, DHR 1972). Following the initial discovery, no further archaeological investigations took place until the 1970s when they were taken up by Carl Clausen. Clausen’s investigations confirmed
bones on the lower slope and fossil remains of extinct mammals on ledges located within the neck of the sinkhole at 21 and 27 meters (Clausen et al. 1979: 609).

Initial investigations in 1971 were followed by the establishment of a research facility in 1975 funded by General Development Corporation and research continued under the direction of Clausen until the early 1980s. This research facility consisted of two residential trailers, three trailers serving as lab, darkroom and offices and also a shed. Many of these original structures are still in use today. In 1982, the spring and a 110-acre buffer zone were donated to the University of Miami for research, which is currently conducted under the direction of Dr. John Gifford (Wentz and Gifford 2007:331).

Little Salt has several important components and at least two occupations: the spring itself, which yielded primarily Paleo-Indian Period artifacts (12,000 to 9,000 b.p.); the slough, which was a middle archaic cemetery; and the midden, an associated middle archaic settlement area immediately to the west of the slough and capping a limestone ridge. Features within the spring can be found along the slope of the basin and also within the neck of the spring. Concentrations of carbonized wood, along with burned bones from deer, bear and alligator, indicate the presence of at least one hearth in the basin and date to about 10,000 b.p., when the water would have been approximately 11 meters below the present surface. Later artifacts, dated to approximately 9,500 years b.p., include a socketed projectile point, base portion of a wooden oak mortar and several hand-fashioned wooden pins that were driven into the sediment near the edge of the opening (FDoS, DHR 1972, Clausen et al. 1979:610). Additionally, the spring contains an unknown number of burials and numerous disarticulated skeletal material between the surface of the spring basin and the drop-off at 13 meters (Wentz and Gifford 2007:331).
The ledge, located approximately 27 meters below the surface is the location of the earliest evidence of human activity. Archaeologists found the shell of a giant extinct land tortoise with a sharply pointed wooden stake between the carapace and the plastron. The positioning of the stake suggests that the implement was used to kill the tortoise. Additionally, long bones and portions of the carapace appear carbonized and were found with fragments of fire-hardened clay, suggesting that the animal was possibly killed and cooked in situ, upside down. Wood from the stake was dated to more than 12,000 years ago (Clausen et al. 1979: 609).

Clausen discovered two partially articulated burials during excavations. The first burial, located in 1986, was found on the west side of the spring in two meters of water. Investigations of this burial revealed that the individual was a young female with intact brain tissue. Testing yielded mitochondrial DNA and radiocarbon dating indicated that the remains date to 6860 ± 110 rcybp (radiocarbon years before present). The second burial was in the northeast quad at a depth of 8 – 9 meters and dated to 5,220 ± 90 rcybp (Clausen et al. 1979:611).

Disarticulated Middle Archaic burials are still present in the spring basin, usually under 5 – 10 centimeters of organic detritus and fresh water biogenic carbonate shell hash. These disarticulated remains are partially mineralized and appear to be from burials interred around the circumference of the spring basin at about 2 meters below the current surface in a freshwater peat deposit. There have been at least five types of archaic cemeteries or burial patterns identified, ranging from midden burials to wet cemeteries. However, the wet cemetery, or mortuary pond, at Little Salt Spring and those akin to it are unique to Florida’s Archaic period. Current research conducted by Dr. John
Gifford and Dr. Rachel Wentz suggests that the similarities between Little Salt Spring and sites like Windover and Republic Groves or Bay West suggest cultural continuity (Wentz and Gifford 2007: 330-331).

From 1992 to the present, underwater excavations conducted by Dr. John Gifford have focused on three, 2 meter by 2 meter test pits in the spring basin, designed to sample the complete stratigraphic section and determine the general depositional sequence in the basin. Concurrent research conducted at the site includes not only studies of the marine life and hydrology, but the analysis of much of the bone recovered in both the scatter and the debris (Wentz and Gifford 2007: 333). Additional research includes sub-bottom sonar survey recently conducted by University of West Florida graduate students and staff to help in the determination of the depositional sequence in the basin of the spring.
CHAPTER III
ARCHAEOLOGICAL WOOD CONSERVATION

Wood is a material that has been used almost universally since antiquity due to its versatility and availability. Thieme cites the discovery of Paleolithic wooden hunting spears dating to 400,000 years b.p. (Thieme 1997:807). As a hygroscopic material, wood is highly sensitive to moisture in its surroundings; therefore, attempts to control the dimensional instability of wood go back nearly as far in time as wood use itself. The loss and gain of moisture can be destructive to wood because of its anisotropic nature (Panshin and De Zeeuw 1964:3). The structural elements of wood tissue are made up of variously shaped and sized wood cells. Each type serves a different purpose, from acting as conduits for moving sap to storing food, and these differing cells are positioned in variable axial positions (Miller 1999:2-3). As these differing cells lose and gain moisture, they experience dimensional changes that are unequal one to the other and can produce problems in wood preservation (Panshin and De Zeeuw 1964:3).

All woods share a certain number of characteristics regardless of type or species. Both hardwoods and softwoods contain the same basic chemical composition and are made up of varying amounts of cellulose, hemicellulose and lignin (Blanchette 2000:189). When wood is subjected to an underwater environment for a substantial amount of time, the cell wall components are weakened through a bacterial action called hydrolysis.
During hydrolysis, the cellulose is decimated, leaving only the lignin network to maintain the original shape of the wood (Watson 1996:9). Over time, water permeates all structures of the wood, filling in the spaces left behind by the deteriorated elements of the wood. As long as waterlogged wood is kept wet, it will retain its shape; however, as soon as it is removed from the environment that is preserving it, the object will begin to collapse as the moisture evaporates. The main considerations when faced with conserving waterlogged archaeological wood are to give the artifact strength to prevent collapse of the object while removing the water and to remove the water without shrinking or distorting the wood (Hamilton 2000:File6). There are varying methods employed to achieve this. Choosing the proper method is dependent on many factors, such as wood species and level of degradation or deterioration as well as the direction of the grain or cut of the wood.

Unger et al. (2001) detail a chronology of wood preservation attempts against insects and moisture going back as far as ca. 4000 BC; however, the most notable attempts at stabilization of waterlogged archaeological wood date to the mid-nineteenth century. In 1861, Danish archaeologist C. F. Herbst published a paper describing his use of the “alum method” to preserve waterlogged archaeological wood. Artifacts were boiled in a supersaturated solution of potassium aluminium sulphate (KAl(SO$_4$)$_2$), or “alum” for two hours and then removed from the solution. The aim was to displace water in the wood cells with a substance that would bulk the cells and prevent them from collapsing as the wood dried (Grattan and Clarke 1987:168). Though this method does not entirely prevent shrinking and often results in heavy, brittle and unnaturally hard
artifacts, it was used on approximately 100,000 artifacts over a period of one hundred years (Unger et al. 2001:6). As working with alum results in less than perfect results and has the additional disadvantage of being difficult to manipulate and dangerous to use due to the use of high temperatures and molten solutions, efforts to determine improved methods for waterlogged wood treatment have been ongoing since the late nineteenth century. However, it was not until the advent of synthetic polymers in the mid-twentieth century that any great developments were made in wood conservation.

Polyethylene glycol, or PEG, was first synthesized in 1859, though it was not commercially produced until almost 1940 in the United States by Union Carbide. PEG is produced in a variety of chemical weights; the lowest weights, 200, 200, 400 and 600 are liquids at room temperature. Mid-range PEGs are soft and waxy and high range PEGs, like 3350, are solids. First applied to use in leather dressings, textile finishing agents, embalming fluids, fabric softeners, and approximately twenty other non-wood applications, PEG was not utilized for the stabilization of waterlogged archaeological wood until almost one hundred years after its first synthesis (Grattan and Clarke 1987:169).

Commercial manufacture of PEG began in the 1940s. In the United States, A.J. Stamm discovered PEG’s ability to prevent shrinkage in wood around the same time that Swedish archaeologists R. E. Moren and K. B. S. Centerwall patented a method of “shrinkproofing” wood using PEG 4000. Initial treatments by Moren and Centerwall using PEG employed an evaporation method, wherein the object was immersed in a heated aqueous solution of PEG 4000. The temperature of the solution was slowly
increased, causing the water to evaporate, eventually leaving the object immersed in pure molten PEG (Grattan 1988:240).

PEG is soluble in ethanol, methanol, and isopropanol, as well as water. Treatment in alcohol is more time efficient and produces more naturally colored objects; however, heating solutions containing alcohol can be dangerous (Hamilton 2000:File 6). In treatment, PEG bulks the wood, slowly displacing the water, so that it may be dried without shrinkage. Bulking can be done through the Moren and Centerwall evaporation method or by placing the object in a solution of lower concentration and slowly raising the concentration. Gradually raising the concentration is important as PEG cannot diffuse into waterlogged wood as rapidly as water is diffused out. The results of simply immersing an object in molten PEG would be unstable as was discovered by Anna Rosenqvist in 1959. Rosenqvist attempted to treat wooden objects from the *Oseberg*, a Viking ship discovered by Norwegian archaeologists in the early nineteenth century, using this method and the results were objects that were twisted, cracked and shrunken (Grattan and Clarke 1987:170).

Experiments attempting to perfect application of PEG to waterlogged wood continued throughout the twentieth century. Woods with more substantial cores were difficult to penetrate with higher molecular weights of PEG eventually leading to a “double-PEG” method in which objects were treated with both a low and a high weight PEG allowing for better penetration of the secondary space in the cell walls and also counteract the effects of cellulose loss in drying (Grattan 1988:247).

In the 1970s, Christensen developed a classification system for waterlogged wood at the National Museum of Denmark as he attempted to treat the Danish Viking ships.
Unable to attain satisfactory results using the evaporation method developed by Moren and Centerwall, he devised the following categories: Category I contained wood with a fairly soft core that was treatable using the Moren/Centerwall method. Category II enveloped those objects with more substantial material and category III includes those objects more difficult to treat. For objects in category III, Christensen developed the tertiary butanol method in which the wood was first dehydrated in this alcohol and then treated with PEG 4000 dissolved in the same solvent. Following impregnation, the artifacts were frozen and then freeze-dried (Grattan 1988:242; Watson 1996:9).

Unlike the impregnation methods discussed in this chapter, freeze-drying was not developed to solve problems of archaeological conservation. It was originally designed by microbiologists to study microorganisms. Progressing studies of microorganisms in the nineteenth century were stalled by the issue of specimen viability over time. Extensive study of biological material was not possible because of this issue. Several attempts were made to store biological specimens by allowing them to air-dry; however, this process caused a loss of virility or activity by the specimens. The development of the liquefaction of atmospheric gases led to a method for freezing biological specimens. After freezing, the specimens were again found to have reduced virility, but it was determined to be because of the thawing process and not the freezing process.

In 1890, R. Altman reported that he was able to dry tissues at subatmospheric pressures under a vacuum at about -20 degrees Celsius. Following his discovery, Benedict and Manning were able to adapt his procedures to dry animal tissues at pressures lower than one atmosphere by means of a chemical pump. This process worked by lowering pressure in the vacuum system by displacing the air with boil-off
vapors of ethyl ether. Once the ethyl ether evaporated, the system was sealed and the residual ethyl ether was absorbed in a separate vessel, which contained concentrated sulfuric acid. During pressure reduction, water vapor was absorbed from the specimen by the concentrated sulfuric acid, reducing the moisture content of the specimen. This pump was inefficient as it took two weeks to reduce the moisture content of the sample to 20% by weight. In 1909, L. F. Shackell replaced the chemical pump with a mechanical pump, like those still in use for vacuum chambers today (Jennings 1999:1-5).

Christensen’s application of freeze-drying was not the first attempt at archaeological wood conservation using this method, but they proved the most successful. Untreated freeze-dried pieces had a tendency to check and crack on the surface. PEG inhibits the formation of damaging ice crystals and further prevents excessive shrinkage of the artifact. After treatment with PEG, the object is frozen. While slow freezing the wood in a commercial freezer would result in large ice crystals oriented in one direction that would improve the rate of freeze-drying, fast freezing using acetone and dry ice produces smaller crystals that are less likely to damage the microscopic structure (Watson 1996:13). The frozen object is placed in a freeze-drying chamber at a temperature of -32° to -40°C. Once the temperature inside the chamber reaches -25°, a vacuum is applied. During freeze-drying, the ice crystals will sublime and all the water will collect on the condenser coil as it is removed (Hamilton 2000:File 6). PEG concentrations used with freeze-drying should be kept well below 50%, as PEG will not freeze at or above that level (Grattan 1988:247).

There are several drawbacks to using PEG to conserve archaeological wood. While it is fairly easy to treat small pieces, large objects require a container or vat large
enough to house the solution and the artifact. Additionally, PEG is more effectively absorbed by the object if the solution is kept heated, which would require further innovation if used on a large object. Beyond logistical and cost considerations, after treatment in PEG, objects usually have a dark, waxy appearance and become unnaturally heavy (Grattan 1988:240). These drawbacks inspired a search for other non-PEG approaches, such as the acetone/rosin method.

The application of the acetone/rosin treatment is generally reserved for waterlogged hardwoods. Gum Rosin, also called colophony, is a solid resin obtained from coniferous trees, primarily pines. Soluble in acetone and alcohol, outside of conservation it is used mostly for soap making, varnishes, sealing wax and adhesives. When an artifact is submerged in an acetone/rosin solution, the acetone displaces the water while the natural pine rosin acts as a bulking agent that replaces the water. This treatment is especially ideal for smaller pieces, producing the most “dimensionally stabilized wood” (Hamilton 2000:File 6). The treatment results in wood that is lightweight, dry, strong and easily repaired through the use of simple adhesives. Additionally, this treatment requires little supervision during most stages of the process. Drawbacks to this method include the cost and its restriction to heartier hard woods. Further, as pine rosin is a natural and not synthesized material, there is a higher chance of impurities or contaminants within the material, and clean up is excessively messy.

The usual procedure for treating an organic artifact with the room temperature acetone/rosin method involves careful preliminary examination and documentation, followed by mechanical and occasionally, chemical cleaning. Once the impregnation is
complete, the samples are removed from the solution and go through a final surface cleaning to remove excess rosin (Fox 1987:81).

Researchers continue to search for new methods for treating waterlogged archaeological wood that are more effective and have more cost and time efficiency. Though one of the most popular treatments for waterlogged wood, polyethylene glycol is both expensive and manufactured from a non-renewable resource. Japanese researchers have recently developed a method of treating waterlogged wood using hydrolyzed keratin from the feathers of ducks. The keratin is used as a bulking agent, much the same way as polyethylene glycol. However, according to Endo et al. (2008), the treatment time is reduced significantly from that required for treating with PEG and the feathers are easily obtainable. However, thus far there has been no documented duplication of this type of process, and it has not been applied to prehistoric wood.

As previously mentioned, very few successful experiments have been conducted to treat prehistoric waterlogged wood. Past conservation treatments applied to artifacts from Little Salt Spring were poorly documented and therefore not easily repeatable. In the late seventies and early eighties, Carl Clausen and his colleagues submitted five proposals to the National Science Foundation (NSF) for funding. Within these proposals, Clausen suggests using one of five methods: (1) alum, (2) white glue, (3) PEG 4000, (4) pine resin and (5) tetraethylorthosilicate.

Of these proposed methods, the only tested methods were the PEG and tetraethylorthosilicate. Tetraethylorthosilicate is a chemical compound with the formula Si(OC₂H₅)₄, often abbreviated TEOS. Applying TEOS in a solution results in a forced petrification, a process not commonly used as a wood conservation method.
Additionally, it is considerably dangerous as exposure to fumes can cause silicosis, a serious disease that causes lesions to form on the lungs. This process was ultimately unsuccessful and when responsibility of conservation of the artifacts moved from Carl Clausen to John Maseman, this treatment was abandoned. Maseman treated several artifacts using PEG and had some success, though apparently the results have questionable stability (Dr. John Gifford, personal communication 2008).

This thesis revisits some of the techniques previously proposed for organic artifacts from Little Salt Spring, specifically, treatment with pine rosin and several different treatments involving the use of PEG. All of the conservation methods employed for this thesis fall into the categories of impregnation and freeze-drying, as expanded below. In addition to techniques using PEG and pine rosin, freeze-drying and impregnation using hydrolyzed keratin were also tested.

Impregnation

The chemical composition of wood consists primarily of two compounds, cellulose and lignin. Cellulose makes up fifty percent of the substance of wood, by weight. During the growth of any given tree, the cellulose molecules are ordered into strands called fibrils, which are further organized into the larger structural elements that make up the cell walls of wood fibers. The amount of lignin varies based on tree type and species. Variation within softwoods is between 23% and 33% and in hardwoods is between 16% and 25%. The lignin resides both within the cell and also between the cells, where it acts as a cementing agent.
Wood cells vary in shape and size as well as orientation. Tracheids, also called wood fibers are cells that are elongated and pointed at both ends with size variation both within the tree and among the species. In softwood species, tracheids are responsible for conducting sap while hardwoods have vessels, also called pores that serve as the main conduits in the movement of sap. Wood rays are oriented horizontally from pith to bark and conduct sap radially along the grain of the wood. The final wood cell type is called the longitudinal, or axial parenchyma cells. The main function of these cells is food storage (Miller 1999:2-3, Grattan 1988:246).

Dry wood cells are generally empty or may be partially filled with gums, resins or tyloses, which are intrusions from parenchyma cells and are made up of protoplasm. The cells in waterlogged wood become deteriorated, a process in which the cellulose is removed, leaving only a soft, weak network composed almost entirely of lignin. The secondary spaces in the cells, or microcapillaries between the strands of cellulose, become enlarged by the loss of material and this is where the water collects (Grattan 1988:246).

Impregnation aims to fill up the spaces in the cellular structure of waterlogged wood with a chemically inert substance that will both give strength and support while the water is being removed. Replacing the water with a synthetic resin prevents the capillary tension effects of simple air-drying, which would lead to collapse. Additionally, filling the cavities of the wood with a bulking agent, or impregnant, provides a moisture-absorbing buffer (Grattan 1988:178). Assessment of wood type and condition are necessary to choose proper bulking agents. Hardwoods are not as easily penetrated by resins with large molecules. The same is true for less deteriorated wood. Penetration
capabilities of the resin increase with increasing deterioration. Undeteriorated wood, or wood that has a considerably hard core, should be consolidated with resins containing smaller molecules such as low weight polyethylene glycol (e.g. 200, 300, or 400). Conversely, resins with smaller molecules cannot be used to consolidate or bulk highly degraded wood because the residual secondary space has become too enlarged. Higher molecular weights must be used to fill the wide spaces (Grattan 1988:247, Watson 1996:15).

Polyethylene Glycol

Polyethylene glycol is synthetically produced polymer made in a range of molecular weights from 200 to 6000. The molecular weight of PEG affects its usefulness in treating certain categories of waterlogged wood. PEG does two things: bulks the cells lumina and infiltrates the cell walls. Where the PEG bulks the wood structure depends on the molecular weight of the substance (Young and Wainwright 1981: 107). Because of the larger spaces in highly deteriorated wood, low molecular weight PEGs, those in the form of liquids such as 200-400, are more or less useless as there is no remaining secondary space for them to occupy. These PEGs are more useful for wood with a more solid core, or category III wood. Wood classified as category II under the Christensen’s classification system is slightly more complicated to treat as it generally exhibits not only a more substantial core but also an enlarged vascular system. Both of these components may be served by treating the wood with the “double PEG” method (Grattan 1988: 247).

When shrinkage occurs in wood, it is not equal in all direction. The least amount of shrinkage takes place along the length of the trunk at 0.1%. Radially, or along lines
from the center outward, shrinkage occurs at 6%. The highest rate of shrinkage happens at the tangential direction, or around the trunk, at 12% (Rosenqvist 1959:20). While treating waterlogged wood with polyethylene glycol, water diffuses out faster than PEG can diffuse in. If the solution does not properly penetrate the object, the impregnation solution will cause rapid dehydration and osmotic collapse (Grattan and Clarke 1987:170). For this reason, it is important to determine the best possible weight of PEG and to gradually raise the percentage of the aqueous solution instead of placing the object in a solution containing a high proportion of the consolidant. Proper molecular weight and the gradual increase of PEG in the solution ensure proper penetration and prevent dehydration, collapse and the twisting and warping that would occur with the unequal shrinkage characteristic of wood cells.

*Colophony*

Colophony is obtained from pines and other conifers. Crude oleo-resin is collected from the trees and processed to make turpentine. In distilling the turpentine, the liquid resin is heated to vaporize the terpene components. The remaining residue is made up of light amber colored, transparent glass-like masses of colophony that can be easily powdered. In archaeological conservation treatments, colophony is most often used in its lump form. It is much heavier than water, but is soluble in naptha, ether, chloroform and acetone (Greenish 1920).

Unlike resins soluble in water, impregnation with colophony, or gum rosin, is reliant on the complete dehydration of the wood. Wood treated with gum rosin must first be completely dehydrated using acetone, as any remaining moisture will impede the
process of impregnation (Grattan 1988:243). Following dehydration, acetone is used as solvent to make up an approximately 67% w/w bulking solution. This solution is moved into the widened spaces by the acetone (McKerrell et al. 1972:112). Unlike synthetic resins, colophony is not manufactured in a variety of molecular weights and is most frequently used in the impregnation of waterlogged hardwoods or wooden artifacts exhibiting less deterioration and the presence of intact heartwood.

**Hydrolyzed Feather Keratin**

Use of keratin as an impregnant for archaeological wood is a new process recently formulated in experiments conducted by Japanese researchers in Kyoto in search of conservation techniques employing renewable resources, primarily as a replacement for the use of PEG. Endo et al. also approached this problem as an environmental issue as treatments using PEG can result in the production of harmful organic acid (Kawahara et al. 2004:93). In addition to presenting problems with waste PEG disposal, development of an organic acid could possibly lead to problems with the stability of the treated wood as well. Researchers working on the continuing treatment and maintenance of the Swedish warship Vasa saw this issue after it was treated with PEG.

The PEG-treated wood was found to contain sulfuric, acetic and formic acids. The sulfuric acid was a result of the ship’s extended contact with the seabed, which contained high levels of sulfur, while the acetic acid is believed to be from the wood itself, as oak is known to have naturally high levels of this type of acid. However, the formic acid may be a result of PEG degradation (Glastrup et al. 2006:22). The molecular break down of the PEG is also worrisome as the structural breakdown will affect the
molecular weight of the PEG, thus changing the melting point and possibly affecting the stability of the wood (Chelazzi et al. 2006:33).

Endo et al. defines keratin as “mammalian epidermal protein complexes established by disulfide cross-linking” and is found in elements of many different animals, including wool, feathers, hair, nails and horns (Endo et al. 2008:1240). Of the different types of keratin, feather keratin was found to be the most suitable and of the tested birds, duck feathers offered the best solution. Feather keratin is a beta keratin that has a lower molecular weight than other keratins, but to use keratin as a conserving agent, it must be hydrolyzed to make a stable solution. This keratin solution was found to be effective on several different species of wood with varying levels of degradation. Also, unlike other impregnants, the hydrolyzed feather keratin does not require as lengthy a treatment time or high percentage solutions (Endo et al. 2008:1240-1245).

**Freeze-Drying**

Freeze-drying is the process of dehydration through sublimation. Water is removed from frozen waterlogged items by turning the frozen water directly into a gas, bypassing the liquid stage. The direct conversion of solid to gas is termed sublimation and for the purposes of lyophilization, or freeze-drying, this process involves the absorption of heat to vaporize the water and the application of a vacuum to more effectively remove the water vapor (Labconco 2004:2).

**Pre-Treatment**

As previously mentioned, impregnation aims to fill up the spaces within the cellular structure of waterlogged wood to lend strength and support while the water is
being removed. The same principle applies to pre-treating artifacts prior to freeze-drying; the presence of a solute protects the wood from freezing damage when ice forms and gives the artifact strength through consolidation. Solutes suggested for pre-treatment include PEG and a number of sugars, including sucrose, sorbitol and mannitol. However, PEG is particularly useful as a pre-treatment method as it has demonstrated an ability to sequester minerals in solution, thus preventing them from crystallizing during freezing and further damaging the structural integrity of the wood’s cellular structure. Further, low molecular weight (liquid) PEG is thought to chemically bond with cellulose, making it less susceptible to changes in relative humidity, while high molecular weight (solid) PEG provides consolidation to degraded areas (Watson 1996:12).

Again, highly deteriorated wood should not be treated using low molecular weight PEG, as there is no residual secondary space remaining for it to occupy. Likewise, high weight PEG will not be able to penetrate the cell walls of less deteriorated wood (Grattan 1988:247). The behavior of PEG relative to its molecular size is the reason it is important to determine the level of deterioration prior to treatment.

**Pre-Freezing**

Proper per-freezing must be carried out in order to have a successfully freeze-dried object. Sublimation cannot occur unless all the solvent is in a solid state, if improperly frozen, the liquid that remains will not be sublimed and if not otherwise removed can be damaging to the object. There are two ways to freeze an object in preparation for freeze-drying: fast freezing and slow freezing. The rapid cooling that occurs when an object is “fast frozen” results in small ice crystals which are useful in
preserving microscopic structures within the wood, but make the object more difficult to freeze-dry. Slow freezing produces large crystals which result in less restrictive channels in the matrix of the object, making freeze-drying more efficient. However, large crystals do little to maintain remaining cellular structure in the wood (Labconoco 2004:4; Watson 1996:13).

The object to be pre-frozen, and subsequently freeze-dried, is placed in a solution made up of a solvent, or the water, and a solute, which is the material dissolved or suspended in the water. Together, the solvent and the solute form a eutectic mixture containing both water and substances that freeze at a lower temperature than water. During cooling, the water will freeze and separate from the solute which will give the appearance that the object is frozen. However, the object is only properly frozen when a temperature is reached at which all of the eutectic mixture is frozen. This temperature is called the eutectic temperature. If an object is not cooled to below the eutectic temperature before freeze-drying commences, tiny pockets of unfrozen material remaining in the object can expand and compromise the structural integrity (Labconco 2004:4; Kramer et al. 2002:1-3).

*Primary Drying*

Primary drying is the first phase of the freeze-drying treatment process that actually takes place in the freeze-dryer. In order for this phase of freeze-drying to be successful, two things must be controlled: temperature and pressure. These two things are related within the vacuum chamber, the higher the temperature of any given object, the higher the pressure. The water molecules within the vacuum chamber have a natural
affinity to move from a high pressure to a low pressure area. The rate of sublimation from the frozen object, therefore, depends on the vapor pressure of the object versus the vapor pressure of the ice collector so the product temperature must be warmer than the collector temperature.

Also important for the success of the primary drying phase is the application of heat in order to vaporize the water, in this case through the use of heated shelves within the vacuum chamber. The heat for sublimation is absorbed through one side of the object, travels through the matrix and vaporizes water on the opposite surface (Ambrose 1975:75/8/4-5; Labconco 2004:5; Labconco 2007:4). In summation, heat travels through the object from the heating shelf to vaporize the water, the vacuum pump expedites the removal of the water vapor from the surface of the object, the difference in temperature and pressure draws the water vapor to the collector and the heat is removed due to ice formation on the collector through the use of a refrigeration system.

Determining drying endpoints for the primary drying stage of freeze-drying has been the subject of many papers and discussions and the miscalculation of said endpoint is frequently the reason for the collapse of freeze-dried objects. The disappearance of visible ice on the object indicates only that the drying of the edges is complete and, as the interior of the object cannot be monitored, the end of primary drying must be determined through other means. This issue will be further addressed in the experiment discussion section of this thesis, as it was a primary concern that affected the outcome of treatments employed in Group I and Group II experiments.
Secondary Drying

Secondary drying is the final drying stage during freeze-drying and generally takes one-third to one-half the time as primary drying. Following sublimation of all ice, and therefore the completion of primary drying, the residual moisture content of the object can still be as high as 7-8%. The object must be dried at a warmer temperature in order to reduce this residual moisture, this is called isothermal desorption. During isothermal desorption, no elevated pressure is required, though the vacuum is not released. Also, while secondary drying requires the application of heat, the amount of heat applied must be compatible with the sensitivity of the object (Kramer et al. 2002:2; Labconco 2004:5).

The following chapter discusses the three Groups that use the conservation methods discussed in this chapter. The experimental treatments used in Groups I, II, and III all contained procedures using some form of impregnation and many also employ freeze-drying, as previously mentioned. Following the discussion of each experiment in the subsequent chapter will be a discussion of the results and recommendations for further treatment of wooden artifacts and/or ecofacts from this site.
CHAPTER IV
RESEARCH DESIGN

Research conducted for this thesis sought to explore, synthesize and evaluate common conservation practices for waterlogged wooden objects, specifically to discover a sound and replicable method to meet the needs of waterlogged wooden artifacts recovered from Little Salt Spring. This prehistoric spring site maintains a constant temperature and contains a high concentration of dissolved minerals while containing almost no oxygen. Previous conservation treatments conducted on organic materials from this site were sparse and poorly recorded. Additionally, some of the techniques previously employed, such as alum and TEOS, have proven to be dangerous or outdated, as discussed in chapter 3. To determine proper conservation methods, experiments were conducted on wood and/or wooden artifacts recovered from both historic and prehistoric contexts. Complementary research was done on the properties of wood, previous experimentation in archaeological conservation, and the site at Little Salt Spring.

Experiments were conducted in three groups. Group I experiments were designed to test methods commonly employed in archaeological wood conservation. Following the completion of experiments in Group I, and analysis of the results, Group II experiments were designed to repeat experiments yielding a successful result, to test for reliability, and alter failed experiments in an effort to achieve successful results. Group II experiments were designed to alter and repeat experiments originally designed for Group
I (Figure 3). Group III experiments were designed to test a method new to archaeological conservation in an attempt to assess its value for treating extremely fragile prehistoric wood like that recovered at Little Salt Spring.

Experiments in Group I were conducted on two pieces of wood that were described as being “average wooden ecofacts” (Dr. John Gifford, personal communication 2008). These two fragments of wood, designated 0911ZW07 and 09108W06 were recovered from Little Salt Spring in June of 2005 from about 8.5 meters below the current surface of the spring in Locus Z. Locus Z designates a unique brown calcite-rich organic mud, which is very dense and considered to have good preservation potential for organic material. Artifacts recovered from this Locus included a worked deer antler fragment with twenty-eight short parallel incisions along its convex edge. A $^{14}$C date was obtained from a wooden ecofact, designated 0910ZW10, recovered about 10 cm away from this antler; it is 9240+/-60 rcybp (radiocarbon years before present). Like the two ecofacts used for this group of experiments, 0910ZW10 was not an artifact, but was collected because it was located close to an artifact. All artifacts and ecofacts from this Locus are considered to be approximately the same age (John Gifford, personal communication 2008). The species of these wooden artifacts are not known, however, the overall site is dominated by Myrica sp. (wax myrtle), Quercus sp. (Oak), Pinus sp. (Pine) and Carya sp. (Hickory) trees. Preliminary examination of the wood samples for the purpose of typing the species suggest that specimen 0911ZW07 is a hardwood – the appearance akin to Salix sp. (Willow), Populus sp. (Aspen or Cottonwood) while specimen 09108W06 is a resinous softwood, perhaps a pine, such as Pinus banksiana
Figure 3. Flow chart exhibiting the relationship between Group I and Group II experiments.
Prior to being subjected to the experimental treatments, each of these two specimen was carefully documented and photographed and then cut into an equal number of pieces so that identical conservation methods could simultaneously be conducted on both. The softwood specimen was comprised of one large piece approximately 16 cm in length and five cm in width. It was difficult to distinguish any detail in the cross section before it was cut into samples as the specimen was spongy. The hardwood specimen was comprised of two pieces; the smaller 11.8 cm in length, and the larger 17.4 cm (Figure 4). A triangular stain at the cross section of each piece indicated where the two pieces were originally joined, making a specimen with the overall length of 29.2 cm. The cut samples from the softwood artifact were mostly uniform at approximately 2 cm thick. However, the hardwood artifact was too fragile to be extensively handled resulting in dissimilar sample sizes (Table 1).

Experiments conducted for Group II were performed on a wooden ecofact also recovered at Little Salt Spring, designated 06280W10, which was cut into seven segments in order to repeat the more successful procedures from Group I in addition to a number of altered procedures based on those initial experiments (Figure 5). Like 0911ZW07 from Group I, this specimen was also a hardwood (Table 2).

Group III experiments were not tested on archaeological wood from Little Salt Spring, but were instead tested on historical wood also recovered from the Emanuel Point
Figure 4. Group I waterlogged wood, preconservation.
<table>
<thead>
<tr>
<th>Specimen:</th>
<th>0911ZW07</th>
<th>09108W06</th>
</tr>
</thead>
<tbody>
<tr>
<td>Wood Type</td>
<td>HARDWOOD</td>
<td>SOFTWOOD</td>
</tr>
<tr>
<td>Overall Length</td>
<td>29.2 cm</td>
<td>16.0 cm</td>
</tr>
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<td>Overall Width</td>
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<td>4.7 cm</td>
</tr>
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<td>Overall Height</td>
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<table>
<thead>
<tr>
<th>Segment</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length</td>
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<td>5.0 cm</td>
<td>5.4 cm</td>
<td>7.6 cm</td>
<td>5.3 cm</td>
<td>2.4 cm</td>
<td>2.1 cm</td>
<td>1.9 cm</td>
<td>5.3 cm</td>
<td>4.3 cm</td>
</tr>
<tr>
<td>Width</td>
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<td>2.9 cm</td>
<td>1.4 cm</td>
<td>1.4 cm</td>
<td>1.0 cm</td>
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<td>4.7 cm</td>
<td>4.7 cm</td>
<td>4.7 cm</td>
<td>4.7 cm</td>
</tr>
<tr>
<td>Height</td>
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<td>2.2 cm</td>
<td>1.7 cm</td>
<td>1.7 cm</td>
<td>2.3 cm</td>
<td>5.4 cm</td>
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<table>
<thead>
<tr>
<th>Segment Treatment</th>
<th>PEG 1450 and Water</th>
<th>Rosin and Acetone</th>
<th>PEG 3350 and Freeze-dry</th>
<th>PEG 3350 and Water</th>
<th>Not Treated</th>
<th>PEG 1450 and Alcohol</th>
<th>Rosin and Acetone</th>
<th>PEG 3350 and Freeze-dry</th>
<th>PEG 3350 and Water</th>
<th>Not Treated</th>
</tr>
</thead>
</table>

Table 1: Group I Specimens, Pre-Treatment
Figure 5. Group II, 0680W10, waterlogged wood, preconservation segments A - E.
<table>
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<th>Specimen:</th>
<th>06280W10</th>
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<tbody>
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<td>Artifact/Wood Type</td>
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<tr>
<td>Overall Width</td>
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<tr>
<td>Overall Height</td>
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</tr>
<tr>
<td>Segment A</td>
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</tr>
<tr>
<td>Segment B</td>
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</tr>
<tr>
<td>Segment C</td>
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</tr>
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<td>Segment D</td>
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<tr>
<td>Segment E</td>
<td>6.3 cm</td>
</tr>
<tr>
<td>Segment Width</td>
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</tr>
<tr>
<td>Segment Width</td>
<td>3.3 cm</td>
</tr>
<tr>
<td>Segment Width</td>
<td>3.5 cm</td>
</tr>
<tr>
<td>Segment Width</td>
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<tr>
<td>Segment Width</td>
<td>3.4 cm</td>
</tr>
<tr>
<td>Segment Height</td>
<td>2.8 cm</td>
</tr>
<tr>
<td>Segment Height</td>
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</tr>
<tr>
<td>Segment Height</td>
<td>2.8 cm</td>
</tr>
<tr>
<td>Segment Height</td>
<td>2.4 cm</td>
</tr>
<tr>
<td>Segment Height</td>
<td>2.8 cm</td>
</tr>
<tr>
<td>Segment Treatment</td>
<td>Oven –dried</td>
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<tr>
<td>Segment Treatment</td>
<td>Rosin and Acetone</td>
</tr>
<tr>
<td>Segment Treatment</td>
<td>Freeze-dry with double PEG pre-treatment</td>
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<tr>
<td>Segment Treatment</td>
<td>Freeze-dry with PEG 3350 pre-treatment</td>
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<tr>
<td>Segment Treatment</td>
<td>PEG 1450 and Water</td>
</tr>
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</table>

Table 2: Group II Specimens, Pre-Treatment
II shipwreck (Figure 6). Little Salt Spring artifacts were not used because of the highly untested nature of the chosen technique. The artifacts utilized for this process were hardwood and likely from the genus *Carpinus*. Woods from this genus are commonly referred to as hornbeam. Wood and wooden artifacts dating to the Archaic, like those at Little Salt Spring, are far scarcer than the waterlogged wood recovered from the Emanuel Point II. Waterlogged wood, mostly in the form of dunnage, was recovered in abundance from the Emanuel Point II and was not necessarily bound for treatment, specifically for that reason. Therefore, the process was applied to the readily available historic wood and not to the archaic artifacts or archaeological wood recovered from Little Salt Spring (Table 3).
Figure 6. Group III, 8ES3345-0146, waterlogged wood, preconservation: (a) 5; (b) 6-2; (c) 4; (d) 2; (e) 3; (f) 6-1.
<table>
<thead>
<tr>
<th>Specimen: 8ES3345-0146, 2</th>
<th>8ES3345-0146, 3</th>
<th>8ES3345-0146, 4</th>
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<td>Not Recorded</td>
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<td>Overall Width</td>
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<td>Not Recorded</td>
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<tr>
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<td>Hydrolyzed Chitin</td>
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Table 3: Group III Specimens, Pre-Treatment
CHAPTER V

GROUP I EXPERIMENTS

All experiments for this thesis were conducted between February of 2007 and December of 2009. As previously mentioned, experiments in Groups I and II were designed so that the results of Group I led to the design of Group II. Treatments used in Group I, and subsequently in Group II, are very common, fundamentally, in archaeological wood conservation and the aim of the experiments was to find a treatment that best fit the needs of waterlogged wooden artifacts and ecofacts from Little Salt Spring specifically, and possibly related or similar sites from Florida’s Archaic Period.

As discussed in Chapter III, Group I experiments were conducted on two separate wooden artifacts designated 0911ZW07 and 09108W06, the former being a hardwood while the latter is a softwood. Each artifact was cut into 5 pieces and subject to identical procedures, assigned the letters A-D. The segments designated as E for both ecofacts were not treated, but set aside for wood species identification.

Subgroup A

Samples in sub-group A were treated using a solution of polyethylene glycol (PEG) 1450 and alcohol. Because of the volatile nature of alcohol, PEG solutions that employ alcohol as a solvent were not placed in an oven, but kept at room temperature (average of 25 degrees Celsius). Keeping PEG solutions at room temperature can present
problems as PEG will begin to solidify at this temperature once the concentration reaches 30%. Concentration for the solutions started at 5%, but for both 09108W06A and 0911ZW07A, the PEG 1450 would no longer dissolve into solution once the mixture reached 50%. For the remaining 20% increase, PEG 3350 was substituted, however, the solution never exceeded 60% concentration.

**Subgroup B**

Following dehydration in alcohol and acetone baths, 09108W06B and 0911ZW07B were placed into saturate solutions of rosin in acetone. The objects were left in the solution for six weeks. Like the PEG and alcohol treatment, the acetone and rosin treatment was kept at room temperature. Following the treatment, the wood segments were removed and cleaned of excess rosin with acetone soaked cloths.

**Subgroup C**

Those pieces designated for freeze-drying, 09108W06C and 0911ZW07C, were placed in a 5% PEG 3350 and water solution and kept at approximately 52°C - 56°C in a laboratory oven. Once the solution reached a concentration of 30%, they were frozen in preparation for the freeze-dryer. The hardwood specimen 0911ZW07C was placed in a commercial freezer to implement the slow freezing process, while the softwood specimen 09108W06C was fast frozen in a slurry of acetone and dry ice, which can reach a temperature of -78°C. Both pieces were placed in a Labconco Freezone 6 Benchtop with tray dryers. This particular model of freeze-drying system has an auto setting for freeze drying applications that was the setting used for these two artifacts.
Subgroup D

The final treatment in Group I also involved the use of PEG 3350. Each specimen was placed into a 5% solution of PEG 3350 and water and kept at approximately 52°C - 56°C as the concentration was slowly raised by increments of 5%. When the solutions reached 70% concentration, the objects were removed and excess wax was wiped away using cloths, Q-tips and dental tools.

Results

For each experiment (A-D), the result was consistently successful or unsuccessful on both specimens, regardless of wood type (Tables 4, 5, and 6). The PEG and alcohol experiments, 0911ZW07A and 09108W06A, were failures. This method resulted in specimens exhibiting three-dimensional failure. The pieces turned dark and waxy, which is not uncommon when impregnating objects using PEG; however, the objects were also shrunken and warped. Both specimens suffered almost a 50% weight change and a comparable dimensional change (Figure 7).

The freeze-drying experiments, 09108W06C and 0911ZW07C, also produced unsatisfactory results. Both specimens appeared very dry and desiccated following treatment, experiencing both splitting and cracking (Figure 8).

Of the finished specimens, the PEG and water treated wood, 09108W06D and 0911ZW07D, proved to be the most dimensionally stable in both woods with no discernible dimensional change, but the treatment resulted in unnaturally dark, heavy and waxy specimens (Figure 9). The acetone and rosin treated specimens were also stable,
<table>
<thead>
<tr>
<th>Group I Specimens</th>
<th>Length Pretreatment</th>
<th>Length Post Treatment</th>
<th>% Change</th>
<th>Width Pretreatment</th>
<th>Width Post Treatment</th>
<th>% Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>09108W06A</td>
<td>2.4cm</td>
<td>2.3cm</td>
<td>4.2%</td>
<td>5.4cm</td>
<td>4.4cm</td>
<td>18.5%</td>
</tr>
<tr>
<td>09108W06B</td>
<td>2.5cm</td>
<td>2.4 cm</td>
<td>4.0%</td>
<td>5.2cm</td>
<td>4.7cm</td>
<td>09.6%</td>
</tr>
<tr>
<td>09108W06C</td>
<td>1.9cm</td>
<td>1.9cm</td>
<td>0.0%</td>
<td>5.1cm</td>
<td>4.8cm</td>
<td>05.8%</td>
</tr>
<tr>
<td>09108W06D</td>
<td>4.3cm</td>
<td>4.1cm</td>
<td>4.6%</td>
<td>5.2cm</td>
<td>5.0cm</td>
<td>03.8%</td>
</tr>
<tr>
<td>0911ZW07A</td>
<td>5.8cm</td>
<td>5.8cm</td>
<td>0.0%</td>
<td>1.7cm</td>
<td>1.6cm</td>
<td>05.8%</td>
</tr>
<tr>
<td>0911ZW07B</td>
<td>5.0cm</td>
<td>5.0cm</td>
<td>0.0%</td>
<td>2.6cm</td>
<td>2.4cm</td>
<td>07.6%</td>
</tr>
<tr>
<td>0911ZW07C</td>
<td>5.4cm</td>
<td>5.3cm</td>
<td>2.0%</td>
<td>1.7cm</td>
<td>1.5cm</td>
<td>11.8%</td>
</tr>
<tr>
<td>0911ZW07D</td>
<td>7.6cm</td>
<td>7.5cm</td>
<td>1.3%</td>
<td>1.7cm</td>
<td>1.7cm</td>
<td>00.0%</td>
</tr>
</tbody>
</table>

**Table 4:** Group I, Percentage of dimensional change in each specimen from pre-treatment to post-treatment

<table>
<thead>
<tr>
<th>Group I Specimens</th>
<th>Wet weight</th>
<th>Dry weight</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>09108W06A</td>
<td>43.6g</td>
<td>25.1g</td>
<td>42%</td>
</tr>
<tr>
<td>09108W06B</td>
<td>45.0g</td>
<td>22.0g</td>
<td>51%</td>
</tr>
<tr>
<td>09108W06C</td>
<td>39.6g</td>
<td>20.0g</td>
<td>49%</td>
</tr>
<tr>
<td>09108W06D</td>
<td>55.7g</td>
<td>51.4g</td>
<td>49%</td>
</tr>
<tr>
<td>0911ZW07A</td>
<td>14.5g</td>
<td>07.1g</td>
<td>51%</td>
</tr>
<tr>
<td>0911ZW07B</td>
<td>30.0g</td>
<td>16.0g</td>
<td>47%</td>
</tr>
<tr>
<td>0911ZW07C</td>
<td>14.7g</td>
<td>06.2g</td>
<td>58%</td>
</tr>
<tr>
<td>0911ZW07D</td>
<td>23.3g</td>
<td>20.8g</td>
<td>11%</td>
</tr>
</tbody>
</table>

**Table 5:** Group I, Percentage of weight change in each specimen from pre-treatment to post-treatment
<table>
<thead>
<tr>
<th>Group I Specimens</th>
<th>Color</th>
<th>Texture</th>
<th>Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>09108W06A PEG 1450 and Alcohol</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>09108W06B Acetone-Rosin</td>
<td>2</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>09108W06C PEG 3350/Water and Freeze-dry</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>09108W06D PEG 3350 and Water</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0911ZW07A PEG 1450 and Alcohol</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>0911ZW07B Acetone-Rosin</td>
<td>2</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>0911ZW07C PEG 3350/Water and Freeze-dry</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>0911ZW07D PEG 3350 and Water</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
</tbody>
</table>

Color: “1” – Color appears lighter than natural wood
“2” – Color appears natural
“3” – Color appears darker than natural wood

Texture: “1” – Dry/Coarse – object may exhibit splintering/rough
“2” – Smooth – object is free from projections or uneven surface/not rough
“3” – Waxy – surface of the object feels and/or has the appearance of wax

Strength: “1” – Brittle – object can be easily broken
“2” – Breakable – object can be broken with some force
“3” – Durable – object cannot be broken without great force

Table 6: Analysis of Treatments, Group I
Figure 7. Group I, Subgroup A, PEG 1450 and alcohol, post-conservation.
Figure 8. Group I, Subgroup C, PEG 3350 and freeze-dry, post-conservation.
Figure 9. Group I, Subgroup D, PEG 3350 and water, post-conservation.
resulting in hard, lightweight pieces exhibiting very little distortion (Figure 10). However, while by no means fragile, the PEG and water treated specimens were by far hardier.

The results of these experiments provided information that allowed for more specifically designed and appropriate methods to use for the next group of experiments. The original methods had to be reassessed before work could commence on Group II artifacts. The failure of the freeze-dried samples proved especially disappointing as this process is fairly common in archaeological conservation, and usually successful when used on smaller pieces of archaeological wood. For that reason, the PEG 3350 and water pretreatment followed by freeze-drying experiment was repeated following further research into the process of lyophilization, to rule out operator error. Additionally, the acetone and rosin treatment was repeated to determine its consistent effectiveness. Also, the use of alcohol was abandoned because of an inability to safely maintain heated alcohol solutions.
Figure 10. Group I, Subgroup B, acetone and rosin, post-conservation.
CHAPTER VI

GROUP II EXPERIMENTS

The second group of experiments was conducted mainly on a large section of archaeological wood recovered from Little Salt Spring and numbered 06280W10. However, as part of the further research into freeze-drying, several freeze-drying experiments were conducted on artifacts not associated with Little Salt Spring. Most of the experiments in Group II involved the use of PEG and the freeze-dryer. However, the acetone and rosin procedure was also repeated on a segment of 06280W10.

Further research into the use of freeze-drying waterlogged wood led to questions regarding the varying effects of using different molecular weights of PEG. During lyophilization, ice is removed from the sample through sublimation. This sublimation process occurs through the object’s absorption of heat, which vaporizes the water. The vacuum then removes the water vapor from the surface of the object and deposits it onto a collector. Effective freeze-drying is dependent on three factors: surface area and thickness of the sample, collector temperature and the vacuum obtained. While a greater surface area allows for a greater rate of freeze-drying, an object that is thicker will slow the process down. As the sample is heated, the water must travel through the dried material to reach the surface and sublimate, the thicker the object, the greater the chance that the dried area may collapse. Fast-freezing the objects reduces this risk, as does the use of a pre-treatment, such as PEG. The use of PEG, however, adds the additional
consideration of melting points and collapse temperatures, which varies between the different weights of PEG.

06280W10A

This segment of 06280W10 was not treated; instead it was placed in the convection oven at approximately 52°C and allowed to air dry over the following six days in order to determine the degree of degradation. The item changed dimensionally by more than 20% and broke into more than twenty pieces (Figure 11). Based on weights taken pre and post conservation, or wet and dry, the artifact is 1360.78% water by weight. Also, using segment A to take samples, 06280W10 was determined to be a hardwood.

06280W10B

Segment B was used to repeat the acetone and rosin treatment. Following dehydration in alcohol and acetone baths, the specimen was placed into a saturate solution of rosin in acetone. After four weeks, the wood segments were removed and cleaned of excess rosin with acetone soaked cloths.

06280W10D

The treatment carried out on 06280W10D was a PEG 3350 pre-treat followed by freeze-drying, which is a repeat of experiments conducted on Group I, C artifacts. The item was placed in a 5% solution of PEG and water and the solution was slowly raised by 5% increments to 30% over several weeks. The eutectic temperature for a solution of PEG 3350 and water is 0°C; therefore, once the fragment was properly pretreated with PEG, it had to be frozen to below 0°C. The equilibrium point of dry ice in acetone is

52
Figure 11. Group II, 0680W10A, post oven-dry.
is approximately -78° C, making fast freezing artifacts treated with PEG 3350 in a slurry of acetone and dry ice an acceptable method.

Once frozen, the segment was placed in the freeze dryer and the temperature was lowered to -50° C before the vacuum was applied. As was previously discussed, the product temperature must be higher than the collector temperature so that the water vapor will be drawn from the object to the collector. In this instance, the best temperature for treatment would be -20°.

As soon as the temperature is lowered using the refrigeration unit, the vacuum is applied and primary drying begins. Determining the end of primary drying is difficult, as moisture content cannot be observed visually with the naked eye. Instead, the end of primary drying was determined through the use of the unit’s manometer, or pressure gauge. Beginning pressure was 138 x 10^{-3} mbar at the start of primary drying and rose steadily for nine days. At that point, the pressure equalized around 77 x 10^{-3}, indicating the end of primary drying. Ideally, secondary drying, which requires the application of heat, can be started at the eutectic temperature of the sample or artifact. Temperatures were gradually increased by 5-degree increments over six days, until the chamber reached 20° C, or room temperature.

For this segment, the double PEG treatment was employed as a pre-treatment prior to freeze-drying, using a 300 weight PEG to penetrate the less deteriorated spaces and 3350 for the more degraded areas of the wood. To carry out a double PEG treatment, subsequent treatments of the low molecular weight followed by the high molecular
weight PEG are applied. The item was first placed in a 10% solution of PEG 300 and water and raised by 10% increments over several weeks to 30%. As lower weight molecules, PEG 300 can move more easily into the spaces within the wood’s cellular structure and is less likely to cause the warping that can be caused by bringing up PEG concentrations too fast. For the higher weight PEG, the solution was more gradually raised. After being treated with the PEG 300, the item was placed into a 5% solution of PEG 3350 and water, slowly raising the solution by increments of 5% over several months to 30%. Once the solution reached 30%, the item remained in solution for several months before the item was frozen and freeze-drying began.

The melting point for PEG 3350 is 56° C while the melting point for PEG 300 is -11° C, making the eutectic temperature -11° C. For this reason, when primary drying commenced, the warming trays were set below the eutectic temperature at -20° C. Primary drying took approximately twelve days to complete, at which point the temperature was slowly raised by ten-degree increments over the following five days.

Water and PEG 1450 replaced the use of alcohol and PEG as a pre-treatment for this Group II experiment. The object was placed into a 5% solution that was slowly raised in increments of 5% over time. Once the solution reached 30%, the object remained in the solution for several months before being frozen and placed in the freeze-dryer. No heat was applied through the heating trays, as the melting point of PEG 1450 is -46° C.
Results

Group II experiments were designed primarily to test freeze-drying methods on organic materials, though the rosin experiment was also repeated. The results were variable, apparently based on differing PEG pre-treatment applications (Tables 7, 8, and 9).

The repeated acetone and rosin treatment, 06280W10B, was conducted in the same manner as B specimens in Group I, however, unlike 09108W06B and 0911ZW07B, this experiment did not result in a hard, naturally colored, lightweight piece, but instead split lengthwise down the sample and distorted. Also, the piece was darkened and tacky to the touch, even after being cleaned with acetone soaked cloths (Figure 12). Again, this segment was treated in the exact same way as the specimens from Group I and was also treated with rosin from the same batch, leading to the conclusion that the result was based on either the type of wood or the direction in which it is cut. Because of the differing size, shape and organization of the wood cells, wood cut in different directions relative to the wood cells may affect the woods’ drying properties.

The specimen freeze-dried following a PEG 3350 pre-treatment, 06280W10D, was the first experiment to use the heating trays as part of the primary drying phase and the heat may have been applied inappropriately. The result was an item that exhibited a hardened side, which may have been burned prior to being waterlogged, while the rest was brittle and was both white and waxy. Over time, the piece began to reveal fine cracks throughout (Figure 13). Specimen 06280W10D was actually put through the freeze-drying procedure prior to the freeze-drying of 06280W10C and helped to determine the proper procedure for applying heat during primary drying.
Table 7: Group II, Percentage of dimensional change in each specimen from pre-treatment to post-treatment

<table>
<thead>
<tr>
<th>Group II Specimens</th>
<th>Length Pretreatment</th>
<th>Length Post Treatment</th>
<th>% Change</th>
<th>Width Pretreatment</th>
<th>Width Post Treatment</th>
<th>% Change</th>
</tr>
</thead>
<tbody>
<tr>
<td>06280W10A</td>
<td>9.7 cm</td>
<td>7.0 cm</td>
<td>27.8%</td>
<td>3.5 cm</td>
<td>1.4 cm</td>
<td>60.0%</td>
</tr>
<tr>
<td>06280W10B</td>
<td>10.8 cm</td>
<td>10.6 cm</td>
<td>1.9%</td>
<td>3.4 cm</td>
<td>2.8 cm</td>
<td>17.6%</td>
</tr>
<tr>
<td>06280W10C</td>
<td>10.0 cm</td>
<td>10.0 cm</td>
<td>0%</td>
<td>3.5 cm</td>
<td>3.0 cm</td>
<td>14.3%</td>
</tr>
<tr>
<td>06280W10D</td>
<td>6.3 cm</td>
<td>6.0 cm</td>
<td>4.8%</td>
<td>3.5 cm</td>
<td>3.3 cm</td>
<td>5.7%</td>
</tr>
<tr>
<td>06280W10E</td>
<td>7.0 cm</td>
<td>6.2 cm</td>
<td>11.4%</td>
<td>3.5 cm</td>
<td>2.4 cm</td>
<td>31.4%</td>
</tr>
</tbody>
</table>

Table 8: Group II, Percentage of weight change in each specimen from pre-treatment to post-treatment

<table>
<thead>
<tr>
<th>Group II Specimens</th>
<th>Wet weight</th>
<th>Dry weight</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>06280W10A</td>
<td>74.5 g</td>
<td>05.1 g</td>
<td>93%</td>
</tr>
<tr>
<td>06280W10B</td>
<td>94.6 g</td>
<td>43.0 g</td>
<td>45%</td>
</tr>
<tr>
<td>06280W10C</td>
<td>78.2 g</td>
<td>25.8 g</td>
<td>67%</td>
</tr>
<tr>
<td>06280W10D</td>
<td>33.5 g</td>
<td>11.4 g</td>
<td>66%</td>
</tr>
<tr>
<td>06280W10E</td>
<td>52.7 g</td>
<td>16.2 g</td>
<td>69%</td>
</tr>
</tbody>
</table>

Table 7: Group II, Percentage of dimensional change in each specimen from pre-treatment to post-treatment

Table 8: Group II, Percentage of weight change in each specimen from pre-treatment to post-treatment
<table>
<thead>
<tr>
<th>Group II Specimens</th>
<th>Color</th>
<th>Texture</th>
<th>Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>06280W10A Oven Dry</td>
<td>3</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>06280W10B Acetone-Rosin</td>
<td>3</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>06280W10C PEG 300/3350/Water and Freeze-dry</td>
<td>2/1*</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>06280W10D PEG 3350/Water and Freeze-dry</td>
<td>1</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>06280W10E PEG 1450/Water and Freeze-dry</td>
<td>3</td>
<td>3</td>
<td>2</td>
</tr>
</tbody>
</table>

Color: “1” – Color appears lighter than natural wood  
“2” – Color appears natural  
“3” – Color appears darker than natural wood

Texture: “1” – Dry/Coarse – object may exhibit splintering/rough  
“2” – Smooth – object is free from projections or uneven surface/not rough  
“3” – Waxy – surface of the object feels and/or has the appearance of wax

Strength: “1” – Brittle – object can be easily broken  
“2” – Breakable – object can be broken with some force  
“3” – Durable – object cannot be broken without great force

* Segment is mostly natural in color, but does have two lighter colored areas on one side at either end.

Table 9: Analysis of Treatments, Group II
Figure 12. Group II, 06280W10B, acetone and rosin, post-conservation.
Figure 13. Group II, 06280W10D, PEG 3350 and freeze-dry, post-conservation.
Inspection of the double-PEG and freeze-dried treated specimen, 06280W10C, revealed that the wood had suffered almost no dimensional loss whatsoever, varying by no more than three millimeters in any direction (Figure 14). Further, unlike the equally dimensionally sound segments treated with only PEG 3350 and water in Group I, the item was lightweight and light colored, not featuring any of the waxy appearance typical of PEG impregnated objects.

The final treatment in this group, applied to specimen 06280W10E, did not produce results useful for analysis. During the freeze-drying process of this particular object, this experiment was interrupted by a power failure. Consequently, the results of this experiment could not be used to determine if this is a useful conservation method (Figure 15).
Figure 14. Group II, 06280W10C, double PEG and freeze-dry, post-conservation.
Figure 15. Group II, 06280W10E, PEG 1450 and freeze-dry, post-conservation.
CHAPTER VII

GROUP III EXPERIMENTS

Experiments in Groups I and II were designed specifically to build upon the success or failure of previous experiments. As noted, successful experiments in Group I were repeated in Group II without alteration, while experiments in both Group I and Group II that failed were altered in an attempt to produce a successful outcome.

Experiments conducted as part of the third group were, in effect, unrelated to the previous experiments in either Group I or Group II, but were instead designed to test the viability of a brand new method of wood conservation introduced by Japanese researchers in 2008.

Using the model suggested by Endo et al., several experiments were conducted in an attempt to recreate their successes (Endo et al. 2010:1313-1315). These experiments may have been complicated by the fact that no feathers could be obtained that had not first been treated, or “washed” chemically for commercial use. Experiments were again conducted on several pieces of wooden dunnage, retrieved from the EPII, artifact number 8ES3345-0146.

8ES3345-0146, Experiments 1, 2, and 3

Experiment 1 was almost immediately abandoned as the hydrolyzation of the feathers failed. For experiments 2 and 3, ten grams of feathers were processed in a solution of sodium hydroxide and then used as an impregnant by mixing the keratin
with water and immersing the wood in the resulting solution. The original concentration was 10% and was slowly raised over a period of two weeks to a concentration of 40%.

8ES3345-0146, Experiment 4

For the fourth experiment, ten grams of waterfowl feathers were pulverized and placed into a solution of sodium hydroxide, or lye, for three hours at 70°C before being neutralized using glacial acetic acid. The resulting solution of hydrolyzed keratin was only about 100 mL. A small piece of wood was placed in a 10% solution of the hydrolyzed keratin and water, and the concentration was raised in 10% increments over the following two weeks to a concentration of 40%.

8ES3345-0146, Experiment 5

The same procedures were followed in this experiment as in experiment 4, concerning the procurement of hydrolyzed keratin from waterfowl feathers. However, instead of gradually raising the concentration of the water/hydrolyzed keratin solution from 10% to 40% using 10% increments over several days, the item was placed directly in a 40% solution and placed in the vacuum chamber under vacuum for one hour.

8ES3345-0146, Experiment 6

The final experiment in Group III was an attempt to discover if other animal derived resources could be used to produce useful bulking agents for the conservation of waterlogged wood. For this experiment the feathers were replaced with squid pens. Keratins are naturally occurring proteins found in the hair, nails, hooves, scales, shells, feathers, beaks and claws of many animals and humans. Squid pens, however, are made
of chitin, a structurally similar substance that is equally tough. Instead of being a protein, however, chitin is a carbohydrate. In some cases, chitin and keratin are combined to form tough material, such as the shells and setae of brachiopods. It was for this similarity that chitin was chosen to test for usefulness as an impregnant for waterlogged archaeological wood.

The same procedure was used to process the squid pens as had been employed for the duck feathers. The squid pens were acquired directly from the cephalopods and ten grams were pulverized and placed in a sodium hydroxide bath for three hours at 70°C. The mixture was then neutralized using glacial acetic acid, after which a small portion was added to water to make a 10% hydrolyzed chitin solution which was to be increased by 10% increments to 40% over two weeks.

Results

The overall results of this particular treatment method for this project proved variable dependent on a variety of factors as discussed below (Tables 10, 11 and 12). The first three experiments accomplished nothing more than opportunity to learn, through trial and error, the proper way to process and hydrolyze the feathers for the treatment. The first and second attempts at hydrolyzing the duck feathers were both unsuccessful; the third attempt did not produce enough of the hydrolyzed keratin to accomplish the final percentage increase and achieve a 40% solution. The first solution was discarded, but experiments 2 and 3 were taken to conclusion regardless and resulted in dry and shrunken pieces (Figure 16).
<table>
<thead>
<tr>
<th>Group III Specimens</th>
<th>Length Pretreatment</th>
<th>Length Post Treatment</th>
<th>% change</th>
<th>Width Pretreatment</th>
<th>Width Post Treatment</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>8ES3345-0146 (2)</td>
<td>08.4 cm</td>
<td>6.8 cm</td>
<td>19.0%</td>
<td>1.9 cm</td>
<td>0.7 cm</td>
<td>63.2%</td>
</tr>
<tr>
<td>8ES3345-0146 (3)</td>
<td>11.7 cm</td>
<td>8.3 cm</td>
<td>29.1%</td>
<td>3.2 cm</td>
<td>1.2 cm</td>
<td>62.5%</td>
</tr>
<tr>
<td>8ES3345-0146 (4)</td>
<td>6.6 cm</td>
<td>5.9 cm</td>
<td>10.6%</td>
<td>3.9 cm</td>
<td>2.4 cm</td>
<td>38.5%</td>
</tr>
<tr>
<td>8ES3345-0146 (5)</td>
<td>10.5 cm</td>
<td>7.6 cm</td>
<td>27.6%</td>
<td>2.3 cm</td>
<td>1.8 cm</td>
<td>21.7%</td>
</tr>
<tr>
<td>8ES3345-0146 (6-1)</td>
<td>10.9 cm</td>
<td>9.2 cm</td>
<td>15.6%</td>
<td>3.6 cm</td>
<td>2.1 cm</td>
<td>41.7%</td>
</tr>
<tr>
<td>8ES3345-0146 (6-2)</td>
<td>10.2 cm</td>
<td>9.1 cm</td>
<td>10.8%</td>
<td>2.4 cm</td>
<td>0.6 cm</td>
<td>75.0%</td>
</tr>
</tbody>
</table>

Table 10: Group III, Percentage of dimensional change in each specimen from pre-treatment to post-treatment

<table>
<thead>
<tr>
<th>Group III Specimens</th>
<th>Wet weight</th>
<th>Dry weight</th>
<th>% change</th>
</tr>
</thead>
<tbody>
<tr>
<td>8ES3345-0146 (2)</td>
<td>15.2 g</td>
<td>2.0 g</td>
<td>87.0%</td>
</tr>
<tr>
<td>8ES3345-0146 (3)</td>
<td>22.2 g</td>
<td>3.9 g</td>
<td>82.4%</td>
</tr>
<tr>
<td>8ES3345-0146 (4)</td>
<td>23.7 g</td>
<td>6.2 g</td>
<td>73.8%</td>
</tr>
<tr>
<td>8ES3345-0146 (5)</td>
<td>10.6 g</td>
<td>2.9 g</td>
<td>72.6%</td>
</tr>
<tr>
<td>8ES3345-0146 (6-1)</td>
<td>15.9 g</td>
<td>5.5 g</td>
<td>65.4%</td>
</tr>
<tr>
<td>8ES3345-0146 (6-2)</td>
<td>07.9 g</td>
<td>2.0 g</td>
<td>74.7%</td>
</tr>
</tbody>
</table>

Table 11: Group III, Percentage of weight change in each specimen from pre-treatment to post-treatment
<table>
<thead>
<tr>
<th>Group III Specimens</th>
<th>Color</th>
<th>Texture</th>
<th>Strength</th>
</tr>
</thead>
<tbody>
<tr>
<td>8ES3345-0146, 2 Hydrolyzed Keratin</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>8ES3345-0146, 3 Hydrolyzed Keratin</td>
<td>3</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>8ES3345-0146, 4 Hydrolyzed Keratin</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>8ES3345-0146, 5 Hydrolyzed Keratin</td>
<td>3</td>
<td>3</td>
<td>2</td>
</tr>
<tr>
<td>8ES3345-0146, 6-1 Hydrolyzed Chitin</td>
<td>2</td>
<td>1</td>
<td>2</td>
</tr>
<tr>
<td>8ES3345-0146, 6-1 Hydrolyzed Chitin</td>
<td>2</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>

Color: “1” – Color appears lighter than natural wood  
“2” – Color appears natural  
“3” – Color appears darker than natural wood

Texture: “1” – Dry/Coarse – object may exhibit splintering/rough  
“2” – Smooth – object is free from projections or uneven surface/not rough  
“3” – Waxy – surface of the object feels and/or has the appearance of wax

Strength: “1” – Brittle – object can be easily broken  
“2” – Breakable – object can be broken with some force  
“3” – Durable – object cannot be broken without great force

Table 12: Analysis of Treatments, Group III
Figure 16. Group III, 8ES3345-0146, hydrolyzed keratin, post-conservation: (a) - (b) Experiment 2; (c) - (d) Experiment 3.
Experiment 4 yielded much more stable results than the first three and exhibited a 25% dimensional change. The surface had a dark and waxy appearance, akin to PEG treated items. Additionally, the surface of the item had a greasy texture. Unlike PEG treated items, however, the item is lightweight. Though more successful than the first three experiments, the percentage of dimensional change keeps this from being a successful experiment. Like the item treated in experiment 4, the specimen treated for experiment 5 exhibited darkening and had a greasy, almost wax-like surface and experienced less than 30% dimensional change (Figure 17).

The final experiment conducted in this group used hydrolyzed chitin instead of keratin, as discussed above. The archaeological wood samples did experience some shrinkage, though it was not severe; the real issue was the dry and peeling surface of the items (Figure 18). The results of this test led to the conclusion that chitin is not a good substitute for this type of treatment and that an actual protein will have to be used to achieve success. Though the experiments of Group III did not ultimately produce an ideal result, the near successes of the items in experiment 4 and 5 indicate that further research should be conducted into this treatment method. Ultimate failure of the procedure for this project may have been due to the type of feathers obtained for the experiment. Commercial avian or waterfowl feathers typically used in bedding or household items are commonly treated through chemical washing. This practice may have had an influence on the effectiveness of the conservation treatment. Feathers may have to be harvested directly from the animal or collected in a way that would not require that they be chemically treated. If repeated and successful, this treatment would most likely result in
Figure 17. Group III, 8ES3345-0146, hydrolyzed keratin, post-conservation: (a)-(b) Experiment 4; (c)-(d) Experiment 5.
Figure 18. Group III, 8ES3345-0146, hydrolyzed chitin, post-conservation: (a)-(b) Experiment 6-1; (c)-(d) Experiment 6-2.
objects that have characteristics of PEG-treated wood, i.e. dark coloring and waxy feel, but would not display the heaviness typically seen by artifacts impregnated with wax.
CHAPTER VIII
CONCLUSIONS AND RECOMMENDATIONS

This thesis sought to address the conservation needs of waterlogged wooden artifacts and ecofacts recovered from the inundated prehistoric site at Little Salt Spring. A total of eighteen experiments were conducted in an effort to find an easily replicable conservation method that best fit the needs of these objects. As discussed, the bulk of the experiments were comprised of the experimental treatments conducted for Group I and Group II. The experimental methods chosen for these groups were based on current conventional, or commonly practiced, conservation methods for all types of waterlogged wooden artifacts and ecofacts. Experiments conducted for Group III wood samples were designed to test a nascent method of waterlogged wood conservation and were tested on wood recovered from a historic underwater site, but never applied to waterlogged wood recovered at Little Salt Spring.

Experiments in Group I were applied equally to both a hardwood specimen and a softwood specimen. For each experimental method, two specimens – a hardwood specimen and a softwood specimen – were given the exact same treatment and kept under the same environmental conditions. The results of each Group I experimental treatment yielded the same, or very similar, results regardless of the species or type of wood of the specimen. Therefore, for Group II experiments, the double softwood/hardwood method
of experiment design was abandoned and all experimental treatments were applied only
one time to a segment of the same waterlogged hardwood ecofact.

Two of the initial experimental treatments used in Group I produced favorable
results. The treatment applied to specimens 09108W06D and 0911ZW07D, PEG 3350
and water, produced specimens that were very durable and exhibited little shrinkage.
However, the pieces were also very dark, heavy, and had a waxy texture. The pieces that
this method were tested on had no surface detail that needed to be preserved; however, if
there were some delicate etching or features that needed to be spared during treatment,
they would have likely been obliterated. Over the past two years, as the other
experimental treatments have been conducted, there has been no change at all in the
specimens.

The second treatment that yielded favorable results was applied to the acetone-
rosin specimens 09108W06B and 0911ZW07B. The specimens treated with this method
were naturally colored and smooth and were shown to be no more breakable than a
modern piece of dry wood. The only exception was a single point on the softwood
specimen that was broken before the treatment that, because of the nature of the
consolidant, can be easily repaired using a mild adhesive. Again, over the subsequent
two years following the treatment, the specimens have exhibited little to no change.

Though the results for these two methods (a total of four experiments) produced
favorable results, the primary researcher for the overall archaeology project requested the
option of different methods because of the possibility of surface obliteration using PEG
and the possibility of heightened flammability using acetone-rosin. For these reasons,
several other experiments were developed for Group II using the same basic treatments but with some methodological alterations.

Treatments in Group II were comprised mostly of varying freeze-drying experiments. Specimen number 06280W10C yielded the most STABLE results using a double PEG treatment (successive PEG 300 and PEG 3350 treatments) followed by freeze-drying. The fragment of wood was treated first with a 30% solution of PEG 300 followed by a 30% treatment of PEG 3350. Freeze-drying resulted in a dry, smooth specimen, but also exhibited strange discolorations and crazing on the surface that were perplexing. Further examination of the object led to the conclusion that the entire ecofact, 06280W10, had been burned prior to being waterlogged. It is important to note that this aspect would not have been evident if the object had been treated solely with PEG, as PEG treatments result in dark, heavy and waxy objects.

From these two groups of experiments, a total of 13 experiments, the five just discussed all produced favorable results with continuing stability. However, of these three, the best option may lie in the double PEG and freeze-dry method. A repeat of the acetone-rosin experiment in Group II did not produce the same favorable results for an unknown reason. Also, as previously mentioned, though the PEG 3350 and water treatment produce stable, durable results, the appearance is unnatural and PEG can obscure the surface detail.

For the experimental treatments applied to Group III object, only those applied to specimen numbers four (8ES3345-0146, 4) and five (8ES3345-0146, 5) yielded results hinting at the possibilities of this type of treatment. Dimensionally, specimen number four in this group changed 10.6% lengthwise and 38.5% along the width, while number
five changed 27.6% lengthwise and 21.7% along the width. The items are darker in appearance than natural wood and have a waxy appearance. They are not heavy, like those ecofacts treated with PEG 3350, but they do have a greasy, almost waxy texture. However, the surface detail does not appear to be obscured. It is the conclusion of this particular study that further research should be done into this method and its viability as a treatment method for objects recovered from this site. As discussed in this thesis, proper materials could not be obtained for these experiments (e.g. unwashed duck feathers), but finding proper materials and retesting these experiments would be a worthwhile endeavor.
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Florida Department of State, Department of Environmental Protection


Florida Department of State, Division of Historical Resources (FDoS, DHR)


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