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A Study to Improve Desalination Methodologies for the Ayyubid City Wall, Cairo

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A STUDY TO IMPROVE DESALINATION METHODOLOGIES FOR THE AYYUBID CITY WALL, CAIRO

Judi Ji won Moon

A THESIS

in

Historic Preservation

Presented to the Faculties of the University of Pennsylvania in
Partial Fulfillment of the Requirements for the Degree of

MASTER OF SCIENCE

2002

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To My Family

Acknowledgements

I would like to thank my advisor Dr. A. Elena Charola for her guidance and support. Her knowledge and experience has proved invaluable. I would also like to thank my reader, Frank Matero, for his helpful advice and assistance..

My gratitude also goes to Rynta Fourie, the manager of the Architectural Conservation Laboratory at the University of Pennsylvania, for her great support and friendship.

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Table of Contents

Figures, Tables, and Graphs

CHAPTER I – INTRODUCTION

1.1 Introduction

In 1998, the Aga Kahn Trust for Culture (AKTC) initiated a project to transform the historic dumping ground of Darassa Hills into a ninety-acre park that will serve as a major green space for metropolitan Cairo. As part of this development, the adjacent historic district of al-Darb al-Ahmar, which is considered an important but deteriorating area of Islamic Cairo, will also be preserved.[1]

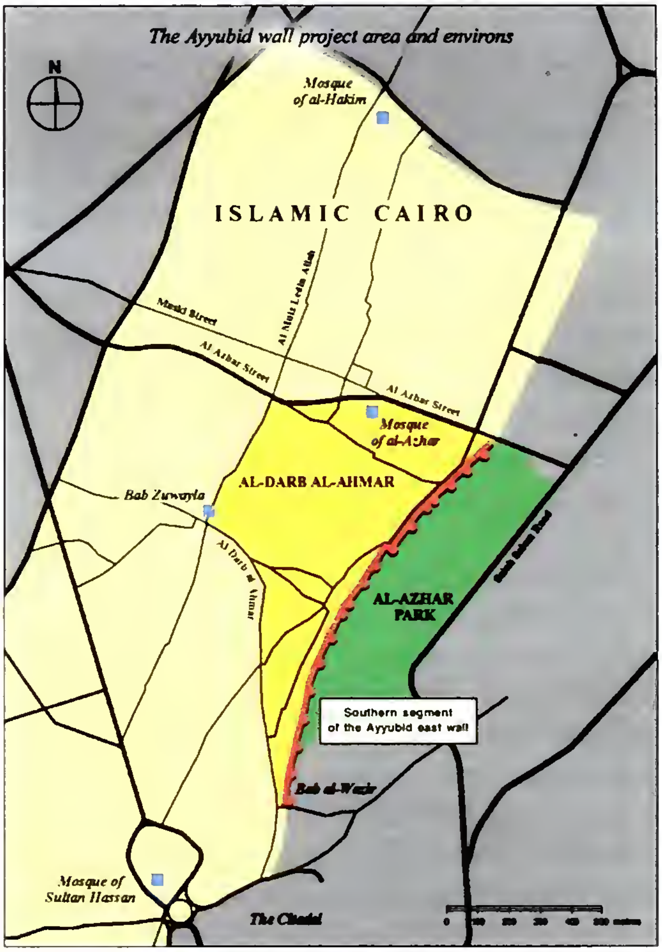

Fig. I.1 The Ayyubid wall project area and environs. *From The Aga Khan Trust for Culture, 2001.*

The Ayyubid city wall, situated along the eastern edge of al-Azhar Park and separating the future park from al-Darb al-Ahmar, is the most notable monument to be addressed in the revitalization plan.[2] The restoration program of the eastern Ayyubid Wall serves as a part of the larger social and physical context of al-Darb al-

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[1] The Aga Khan Trust for Culture, *The Eastern Ayyubid Wall of Cairo: Study Findings and Recommended Conservation Programme*, *Historic Cities Support Programs Technical Brief* no. 4 (2001), p. 3.

[2] ibid.

Ahmar where the aim of the project is to preserve and develop al-Azhar Park and the adjacent residential district.[3]

1.2 Historic Fortifications of Cairo: A Brief Introduction

For more than a millennium, the walls of Cairo witnessed the city's evolution as it changed from a royal precinct to the center of military power, and eventually to a major metropolis. Although the walls have seen perennial changes throughout the centuries, there are several fortification campaigns that can be divided into three main periods of construction.[4]

The Fatimids, who founded the city of Cairo in 969 AD, built the initial enclosure. The walls were made of mud brick, but they have been obliterated over time. The second fortification was built at the end of the eleventh century, also in the Fatimid era, where the period of civil unrest is reflected in the addition of stone gates to the mud brick walls.[5] Vestiges of this campaign can still be seen in the three surviving gates. The third campaign, built in the 12th century by Salā ad-Dīn (the founder of the Ayyubid dynasty) and his successors, is a more complex scheme built entirely of stone. The southern segment of the eastern fortification that forms a boundary between what is to be the new al-Azhar park and the historic district is part of this last campaign.[6]

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[3] The Aga Khan Trust for Culture, *The Eastern Ayyubid Wall of Cairo: Study Findings and Recommended Conservation Programme*, *Historic Cities Support Programs Technical Brief* no. 4 (2001), p. 8.

[4] AKTC, p. 13.

[5] ibid.

[6] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 6.

1.3 The Southern Segment of the Eastern Ayyubid Wall

Constructed as a double limestone veneer with a rubble-mortar core, this portion of Cairo's historic wall stretches for approximately 1,200 meters. It is 3 to 3.5 meters wide, and up to 9 meters high.[7] It has served as the eastern boundary for hundreds of years. However, with the gradual expansion of the city it ceased to serve as a defensive structure, and from the 15th century on, the area just outside the wall began to be used as a dumping ground. The wall was eventually buried under the debris, forming what is now known as the Darassa Hills.[8]

Fig. 1.2 (Top) The western slope of the Darassa Hills in 1924; (middle and bottom) the same view in 1995 and 2001. *From The Aga Khan Trust for Culture, 2001.*

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[7] M. McCormack, *Conservation Studies for the Ayyubid City Wall, Cairo* (master's thesis, University of Pennsylvania, 2001), p. 5.

[8] The Aga Khan Trust for Culture, *The Eastern Ayyubid Wall of Cairo: Study Findings and Recommended Conservation Programme*, *Historic Cities Support Programs Technical Brief* no. 4 (2001), p.21.

By the 18th century, the "mountains of debris" were high enough to offer a strategic view over the city. As a matter of fact, during the Napoleonic campaign of 1798, the French army utilized the Darassa Hills and certain sections of the Ayyubid wall as military posts.[9] In concern for its structure and functionality, the French made what can be called a few "maintenance interventions" to the wall.[10]

Other sporadic repairs followed in the early 20th century with the establishment of the Comité de Conservation des Monuments de l'Art Arabe, a government body organized to preserve Egypt's Islamic and Christian architectural heritage. Although a more involved reconstruction of the two towers and the replacement of missing veneer wall masonry was carried out in 1950, the wall remained buried under the debris as the dumping continued. It was not until 1997, when the AKTC began landscaping the Darassa Hills for the al-Azhar Park that the debris was cleared.[11]

1.4 In Recent Years

In 1999, a comprehensive material condition survey was performed. While the stone conditions ranged from well preserved to irreparably deteriorated, it was determined that widespread features such as flaking and disaggregation of the stone surface were due to intrinsic salt crystallization within the stone. Moreover, different types of salt deposits were found depending on the location on the wall.[12]

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[9] The Aga Khan Trust for Culture, *The Eastern Ayyubid Wall of Cairo: Study Findings and Recommended Conservation Programme, Historic Cities Support Programs Technical Brief* no. 4 (2001), p. 21.

[10] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p 8.

[11] AKTC, p. 21.

[12] M. McCormack, *Conservation Studies for the Ayyubid City Wall, Cairo* (master's thesis, University of Pennsylvania, 2001), p. 14.

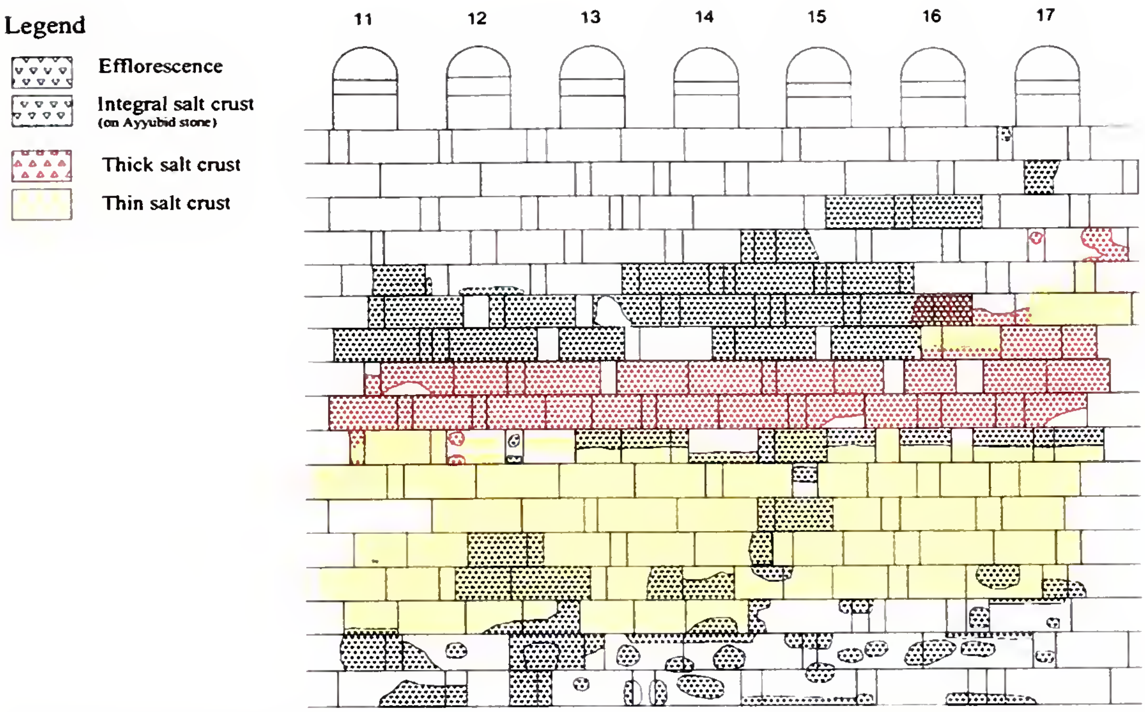

Fig. I.3 Survey of salt deterioration patterns between tower 4 and 5 of the eastern section of the Ayyubid Wall. *From Bourguignon, 2000.*

In order to determine the salt distribution within the wall, four samples of both the original and Comité's 1950 replacement stones were cored at different levels of the wall.[13] In addition, qualitative chemical analysis such as micro-chemical spot-tests, thin section, microscopy, Scanning Electron Microscopy (SEM), and X-Ray Diffraction (XRD) were performed on eleven samples taken from the veneer stone.[14]

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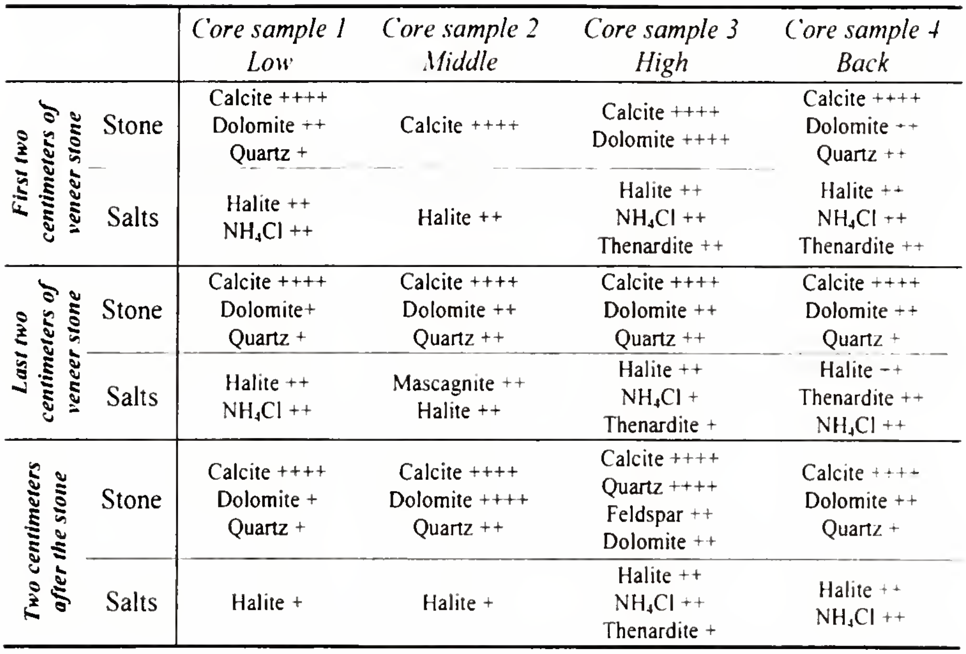
[13] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 101-102.

[14] M. McCormack, *Conservation Studies for the Ayyubid City Wall, Cairo* (master's thesis, University of Pennsylvania, 2001), p. 18.



		Core sample 1 *Low*	*Core sample 2* *Middle*	*Core sample 3* *High*	*Core sample 4* *Back*
First two centimeters of veneer stone	Stone	Calcite ++++ Dolomite ++ Quartz +	Calcite ++++	Calcite ++++ Dolomite ++++	Calcite ++++ Dolomite ++ Quartz ++
	Salts	Halite ++ NH_4Cl ++	Halite ++	Halite ++ NH_4Cl ++ Thenardite ++	Halite ++ NH_4Cl ++ Thenardite ++
Last two centimeters of veneer stone	Stone	Calcite ++++ Dolomite+ Quartz +	Calcite ++++ Dolomite ++ Quartz ++	Calcite ++++ Dolomite ++ Quartz ++	Calcite ++++ Dolomite ++ Quartz +
	Salts	Halite ++ NH_4Cl ++	Mascagnite ++ Halite ++	Halite ++ NH_4Cl + Thenardite +	Halite ++ Thenardite ++ NH_4Cl ++
Two centimeters after the stone	Stone	Calcite ++++ Dolomite + Quartz +	Calcite ++++ Dolomite ++++ Quartz ++	Calcite ++++ Quartz ++++ Feldspar ++ Dolomite ++	Calcite ++++ Dolomite ++ Quartz +
	Salts	Halite +	Halite +	Halite ++ NH_4Cl ++ Thenardite +	Halite ++ NH_4Cl ++

Fig. I.4 Results of the XRD analysis performed on the core samples. *From Bourguignon, 2000.*

Such analyses confirmed that salts were indeed present throughout the wall. SEM revealed that salts had crystallized within the stone's voids, inducing micro-cracks throughout the matrix. XRD displayed the presence of halite (sodium chloride) as the major component, along with ammonium chloride and anhydrous sodium sulfate.[15]

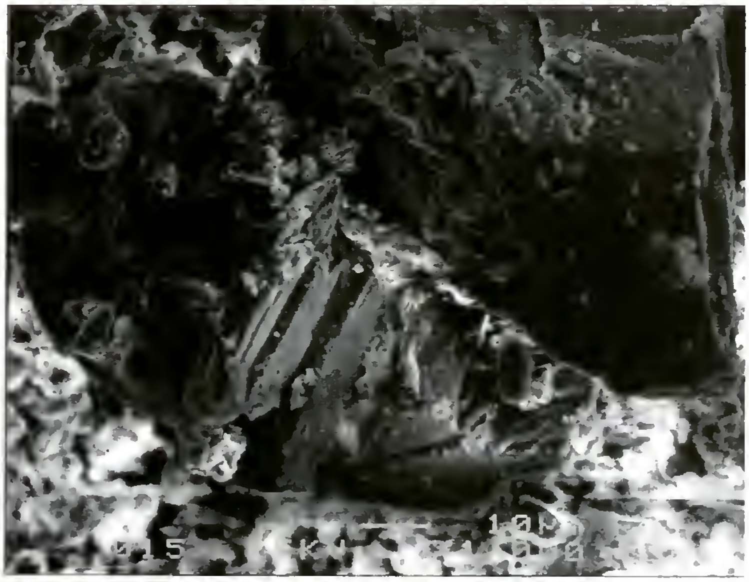


Fig. I.5 Scanning electron micrograph of a surface flake showing salt crystals growing out of a micro-crack. Magnification x1000. *From Bourguignon, 2000.*

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[15] Bourguignon, p. 110.

Based on the results, several treatment methodologies for desalination were evaluated. Given the large scale of the Ayyubid Wall, however, only a few options were considered practical. Possibilities included: consolidation and poulticing as well as the application of sacrificial renders.

In 2000, a thesis by E. Bourguignon evaluated the effects of surfactants in mitigating the expansive properties of the limestone's clays. Due to the strong influence the tested surfactant had on the crystallization habit of NaCl, its effect on improving desalination by poulticing was explored in more detail by M. McCormack in 2001.

1.5 The Current Program

Following the research of the two authors aforementioned, this thesis aims to further analyze the promising effects the surfactants have on desalination. The Egyptian limestone samples to be used for testing are from the same quarry as the currently proposed replacement stones. However, in order to simulate the actual conditions of the wall, the sample stones were first impregnated with the principal contaminating salt (NaCl), followed by accelerated aging. Desalination through the use of more economical surfactants was tested by the application of a non-ionic Triton™ XL-80N surfactant and an anionic Orvus WA paste surfactant.

CHAPTER II – EGYPTIAN LIMESTONE: A Summary of Composition and Deterioration Characteristics

2.1 Composition

Egyptian limestone, one of the major types of building stones used in ancient Egypt, was formed some 80 million years ago by the precipitation of calcium carbonate from seawater and the deposition of silt. During the formation, gypsum (calcium sulfate dihydrate) and halite (sodium chloride) also crystallized, uniting with calcite and sand to form a sedimentary stone composed of shale and clays on the uppermost geological stratum. Egyptian limestone consists mainly of calcite (calcium carbonate) along with other secondary minerals such as clays, quartz, and gypsum to name a few. The relatively high clay content originates from the silt and clay particles settling onto the oceanic floor and becoming incorporated into the limestone.[16]

2.2 Deterioration Characteristics

2.2 a - Salt Crystallization

It has been observed that Egyptian limestone contains a high soluble salt content. This is not surprising since sodium chloride (halite) and gypsum are part of its natural constituents. However, these soluble salts are the primary cause for stone deterioration

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[16] F.M. Helmi, "Study of Salt Problem in the Sphinx, Giza, Egypt," *In Preprints of the 9th Triennial Meeting of the ICOM Committee for Conservation* (Dresden, 1990): 326-329, referenced in E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 20.

due to their repeated dissolution and crystallization cycles.[17] In order to better understand this deterioration process, the relationship between moisture and salt will be briefly discussed.

Salts move through a porous body when dissolved in water. The presence of water in the pores can originate from capillarity, infiltration, condensation, and hygroscopicity. Capillarity and infiltration are mechanisms of liquid water that take place due to the surface tension of water, attractive forces between the water and the pore walls, and hydrostatic pressure. Condensation and hygroscopicity, on the other hand, are mechanisms that introduce water as water vapor. Condensation can occur either on the surface or in the pores as a result of changes in the ambient conditions (temperature and relative humidity), whereas hygroscopicity refers to a process where a material ad- and/or ab-sorbs moisture from the air. In this latter mechanism, the degree of moisture ad/absorbed is dependant on the nature of the porosimetry of the material (pore-size and distribution).[18] Once the water is introduced and is in liquid phase, it can transport salts. In the liquid form, salts are transported through capillary action or by diffusion, moving from higher salinity to lower salinity regions.[19]

Salts can precipitate in two ways. When salts crystallize in an aqueous solution, it is due to supersaturation. The other type of precipitation occurs as a result of a salt-

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[17] K.L. Gauri, "The deterioration of Ancient Stone structures in Egypt," *In Prospection et Sauvetarde des Antiquities de l'Egypte: Actes de la Table Ronde organisée á l'du Centenaire de l'Institut Française d'Archéologie Orientale* (1981): 13-18, referenced in E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 19.

[18] A.E. Charola, "Salts in the Deterioration of Porous Materials: An Overview," *Journal of the American Institute for Conservation* 39 (2000): p. 328.

[19] B.M. Feilden, *Conservation of Historic Buildings* (Boston: Butterworth Scientific, 1982), p. 100.

solution-vapour system.[20] The water vapor pressure over a salt solution is lower than the vapor pressure of pure water at a given temperature. Therefore, there is an inverse relationship between the concentration of the salt solution and the water vapor pressure: as the concentration of the solution increases, the water vapor pressure decreases. When the solution is saturated, the water vapor pressure has reached its lowest value. This is known as the "equilibrium relative humidity" (RH_{eq}) and it varies depending on temperature and the type of salt present in the solution.[21]

If the relative humidity is greater than the salt's RH_{eq}, the environment is humid enough to allow the salt to absorb water vapor from the air and exist as an aqueous solution. For example, NaCl has 76% RH_{eq}. As long as the ambient RH is above 76%, NaCl will tend to exist as a saturated solution rather than a solid.[22] If the relative humidity falls below the salt's RH_{eq}, the solution will become supersaturated, enabling crystallization. Therefore, variations in the relative humidity can produce crystallization/dissolution cycles.

In an arid climate such as the one found in Cairo, the high temperature of the daytime causes the water to evaporate from the stone surface. This generates a concentration of water-soluble salts closer to the surface since it draws the moisture and the salts from the depth of the stone. The evaporation of water decreases the local relative humidity to drop below the RH_{eq} of the salts, promoting their crystallization as explained

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[20] A. Arnold and K. Zehnder, "Salt Weathering on Monuments," *La Conservazione dei monumenti nel bacino del Mediterraneo: Influenza dell'ambiente costiero e dello spray marino sulla pietra calcarea e sul marmo*, Atti del 1° Simposio internazionale Bari 7-10, giugno (1989), p. 47.

[21] A.E. Charola, "Salts in the Deterioration of Porous Materials: An Overview," *Journal of the American Institute for Conservation* 39 (2000): p. 328.

[22] Erhardt and Mecklenburg, "Relative Humidity Re-Examined," *Preventive Conservation, Practice, Theory and Research: In Preprints of the Contributions to the IIC Ottawa Congress* (London: IIC, 1994), p. 34.

above. Conversely, the decrease in evening temperature results in an increase of the relative humidity, inducing moisture to condense as droplets and penetrate into the stone. The water-soluble salts are dissolved in the process whereby the solution enters the pores of the stone through capillary action, only to crystallize again during the heat of the following day. Such dissolution and crystallization cycles create a great deal of stress in the stone, promoting physical deterioration.[23]

A critical factor in the deterioration process is the location of salt crystallization. That is, crystallization takes place at different depths of a stone depending on the rate of water evaporation from the surface and the rate at which the solution is replenished at that location. The former is due to environmental/physical parameters such as temperature, relative humidity, surface area and roughness of the stone. The latter is determined by the physical properties of a salt solution such as its surface tension, vapor pressure, and viscosity.[24]

When the solution travels to the surface at a rate equal to or faster than the rate at which it dries, the solute deposits on the external surface as "efflorescence." However, if the water on the surface evaporates more rapidly than the rate at which fresh water solution migrates to the surface through the capillary system, a dry zone develops just beneath the surface. Solute is deposited within the porous system

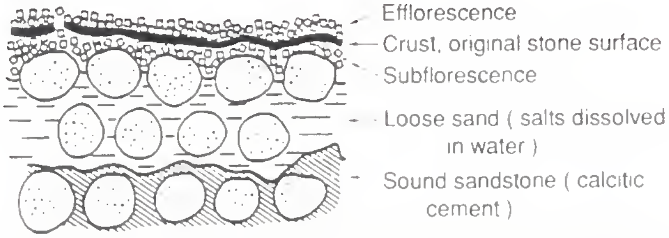

Fig. II.1 Salt efflorescence and subflorescence, a schematized profile across a sedimentary stone. *From E.M. Winkler, 1994.*

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[23] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 35.

[24] C. Rodriguez-Navarro and E. Doehne, "Salt Weathering: Influence of Evaporation Rate, Supersaturation and Crystallization Pattern," *Earth Surface Processes and Landforms* 24, no. 3 (1999), p. 191.

at the boundary between the wet and dry regions of the initial moisture content and referred to as "subflorescence."[25] If the migration of the solution to the surface takes place over a long period of time or if evaporation takes place within the pores of the stone, "cryptoflorescence" [26] results where the salt crystallizes deep within the stone and does not manifest itself in the form of surface decay.[27] While the presence of efflorescence is the most visible, it generally serves as an indication of salt concentration within the stone rather than acting as an agent of deterioration itself. The most extensive salt decay occurs when the solute is deposited within the pores of the solid just beneath the external layer, as in the case of subflorescence.[28]

In addition to the location of salt formation, the habit of the crystallized salt can also play an important role in the type and severity of deterioration induced in the stone. According to theories of crystal morphology, a crystal has the tendency to develop the faces with lowest surface energies according to its crystal structure. If the environment is homogeneous and if slow crystallization is possible, the crystals develop an ideal "equilibrium form." In reality, however, this ideal form is rarely achieved due to changes in the environment. Therefore, salts often crystallize under their "growth forms" or "habits," which differ significantly from their equilibrium forms.[29] Most habits are not

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[25] S. Z. Lewin, "The Mechanism of Masonry Decay Through Crystallization," *Conservation of Historic Stone Buildings and Monuments* (Washington, D.C.: National Academy Press, 1982), p. 120.

[26] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 31.

[27] S. Z. Lewin, "The Mechanism of Masonry Decay Through Crystallization," *Conservation of Historic Stone Buildings and Monuments* (Washington, D.C.: National Academy Press, 1982), p. 122.

[28] C. Rodriguez-Navarro and E. Doehne, "Salt Weathering: Influence of Evaporation Rate, Supersaturation and Crystallization Pattern," *Earth Surface Processes and Landforms* 24, no. 3 (1999), p. 193.

[29] A. Arnold and K. Zehnder, "Crystallization and Habits of Salt Efflorescences on Walls II: Conditions of Crystallization," *The 5th International Congress on Deterioration and Conservation of Stone* (1985), p. 270.

very stable: they can undergo transformations as a process of ageing or by re-dissolution/re-crystallization from further supply of moisture. As such, different salt crystals with different structures can exhibit the same habits. Conversely, the same salt minerals can form different habits at different places depending on external conditions. For example, efflorescence can manifest as "fluffy efflorescence" or as "salt crusts." The first cause relatively minimal damage to the stone since they can easily be brushed off the surface while salt crusts prove more damaging for they are difficult to remove.[30]

Fig. II.2 Both Egyptian limestone cubes were impregnated with NaCl. The cube on the left exhibits both whisker growth and salt crusts, whereas the sample on the right manifests only the salt crusts.

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[30] A. Arnold and A. Kueng, "Crystallization and Habits of Salt Efflorescences on Walls I: Methods of Investigation and Habits," *The 5th International Congress on Deterioration and Conservation of Stone* (1985), p. 256 and 259.

2.2 b - Clay Minerals

Studies have shown that moisture related expansion of the clay minerals is another factor responsible for the damages observed in Egyptian limestone. Water penetration within the pore system of the stone can cause the clays to swell with moisture and shrink with evaporation. Such repetitive action creates damaging pressure to the internal stone structure. There are two types of swelling: inner-crystalline and osmotic swelling.[31] While the swelling behavior depends on conditions such as the type and quantity of clay minerals, the deterioration rate caused by the swelling is largely dependant on the spatial distribution of the clay minerals contained in the stone.

The inner-crystalline swelling commonly occurs in expandable clays such as smectite and vermiculite. This is due to the hydration of the shared cations present between the layers of the dry clay. The dehydrated clays consist of sheets that have a negative charge on the surface and a positive charge on the edge, which are held together by the charge-compensating cations.[32] When the polar water molecules come in contact, the molecules orient their negative poles toward the cations and their positive poles toward the negatively charged surface of the clay, allowing the cations to hydrate. This weakens the electrostatic interaction between the interlayer cations and the negatively charged layers, increasing the inter-layer distance. Crystalline swelling, then, occurs only for the type of clays with exchangeable interlayer cations.

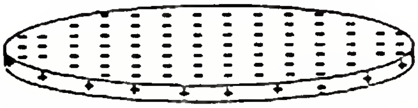

Fig. II.3 Clay sheet with negatively charged surface and positively charged edge. *From Jon Otto Fossum, 1999.*

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[31] Van Olphen, *An Introduction to Clay Colloid Chemistry, 2nd ed.* (New York: John Wiley, 1997).

[32] Jon Otto Fossum, "Physical Phenomena in Clays," *Physica*, 1999 [cited 13 March 2002]; available from URL: http://www.phys.ntnu.no/~fossumj.

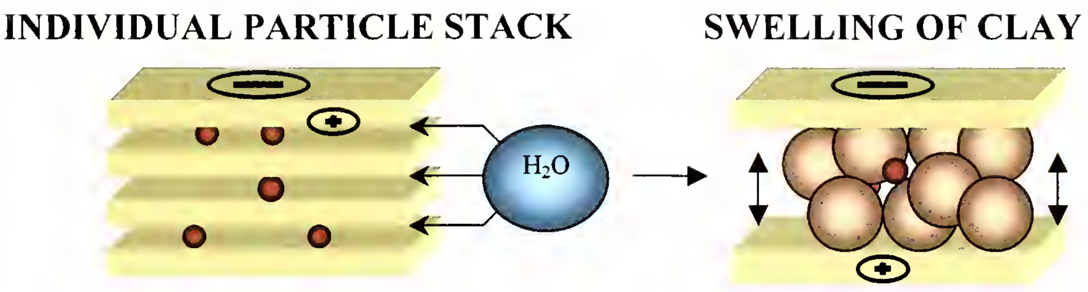

Fig. II.4 Inner-crystalline swelling of clay.

▰ Clay sheet – negative surface, positive charge edge

● Cation - positively charged, pulls the negatively charged clay surfaces together

● Water molecule – positive-end attaches to the negative clay surface while the negative-end attaches to the cation

● Hydrated cation

Adapted from Jon Otto Fossum, 1999.

Although clays considered nonexpandable sometimes experience minor crystalline swelling, most nonexpandable clays such as illite and kaolinite undergo osmotic swelling. This can act over much larger distances than the inner-crystalline swelling, but the swelling stress produced is significantly smaller than that of inner-crystalline swelling.[33] Here, the swelling occurs due to a difference in the ion concentration between the ions close to the clay surface and the ions in the pore water of the rock. Hence, diffusion of the two concentrations results if the salt ion concentration is greater then the pore water

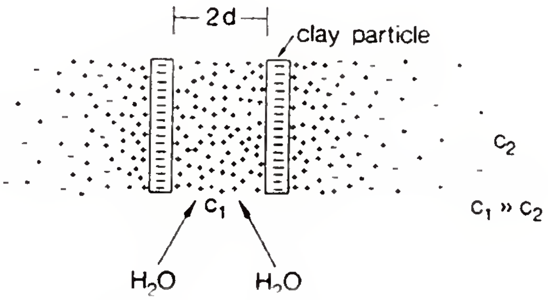

Fig. II.5 Clay layers with ion cloud. *From Madsen and Müller-Vonmoos, 1989.*

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[33] Madsen and Müller-Vonmoos, "The Swelling Behavior of Clays," *Applied Clay Science* 4, no. 2 (1989), p. 147.

concentration, permitting water to penetrate the interlayer space of the clay. The negatively charged surface of the clay, then, adsorbs the polar liquid as well as any other ions present.[34]

As mentioned earlier, clay swelling produces pressure which can damage the internal stone structure. However, it is the spatial distribution of the clay minerals that dictates the rate at which the stone will disintegrate. In order for clays to swell and compromise the physical soundness, they must be accessible to water. In the case of Egyptian limestone, it has been found that the clays are often concentrated along the bedding planes. When tested by partial water immersion and relative humidity cycling, the stone exhibited fractures parallel to the bedding planes. It also displayed spalling and delamination followed by extensive disintegration and permanent mechanical deformation, in a relatively short period of time.[35]

Moreover, some scientists have identified in the deterioration process, a direct relationship between the presence of salts and clay. It has been observed that the swelling of clays is actually enhanced by the presence of salts. Although the complete mechanism has yet to be fully understood, experiments have shown that salt-contaminated stone actually contracts during wetting and expands during drying. That is, the grain-to-grain distances in the clay aggregates are smaller in the wet state than in the dry state. Furthermore, such hygric dilatation is not reversible and the amount of dilatation increases from cycle to cycle. It is believed that contraction during the wet phase is caused by the formation of dense hydration shells between the grains, which become

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[34] Rodriguez-Navarro et al., "The Role of Clays in the Decay of Ancient Egyptian Limestone Sculptures," *Journal of the American Institute for Conservation* 36, no. 2 (1997), p. 162.

[35] ibid.

denser as electrolytes become stronger. Expansion, on the other hand, may be due to salt crystallization in the stone pores or from the salt films that adhere to the grains, pushing the grains apart as they grow. In the initial state, the supply of salt (cations) causes contraction and the supply of water causes expansion. However, as more salt is introduced, the contraction becomes stronger until at constant salt content, the absorption of water causes a decrease in salinity, subsequently causing the system to expand.[36] (Fig. II.6)

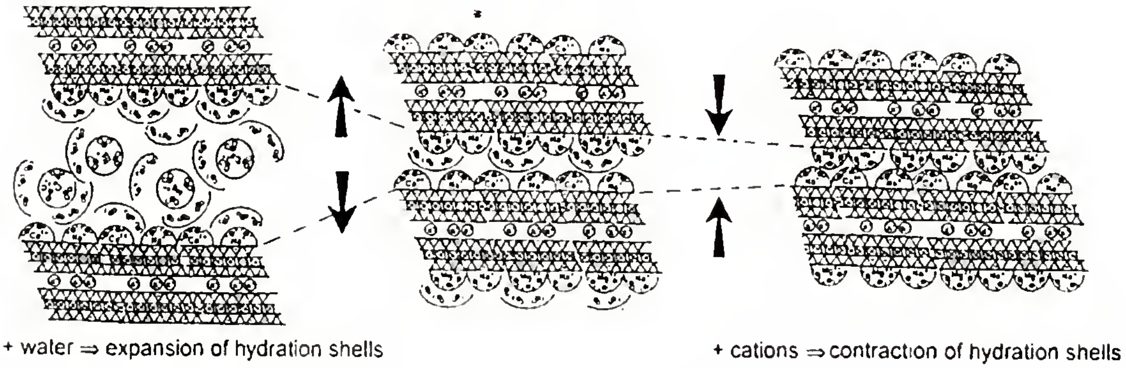

Fig. II.6 Expansion and contraction of clays showing the effect of introducing salts and water to a colloid system of clays. *From Snethlage and Wendler, 1997.*

Also, the presence of salt such as NaCl within the stone offers a supply of sodium ions to the water entering the pore system. The sodium ions, however, become hydrated in turn and produce an initial swelling of single fibrous clay crystals (crystalline swelling). With complete hydration of the clay surface, there forms an electrical double layer. The interparticle swelling (osmotic swelling), then, results due to the electrostatic repulsion forces between nearby clay particles. Even if the clay particles are closely

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[36] Snethlage and Wendler, "Moisture Cycles and Sandstone Degradation," *Saving Our Architectural Heritage: Conservation of Historic Stone Structures* (1997), p. 12-14.

packed, this process can create enough pressure to damage the layered structure of the stone.[37]

Furthermore, the presence of salt promotes condensation of water at RH values lower than 100%. For example, NaCl will deliquesce when RH is above 75%, enhancing the problem due to clay expansion under minimal RH changes. In addition, salt mixtures tend to reduce the RH_{eq} of the salts. Therefore, if NaCl is accompanied by another type of salt (which is expected), liquid water can be present within the stone's pore system even when the RH is below 75%.[38]

Relative humidity cycles lead to water condensation and subsequent clay swelling. By providing ions and reducing the RH_{eq}, the presence of salts contributes to the irreversible damage of the stone structure. Cyclic expansion and contraction of the clays affects the internal microstructure of the stone, such as the change in pore size distribution. Open pores provide easy access for moisture and greater surface area for chemical reaction; therefore porosity and the number of micro-pores can render the stone more vulnerable to deterioration. In fact, stones with a higher proportion of micropores connected to large pores are more susceptible to salt weathering.[39]

In conclusion, three factors are significant to the Egyptian limestone decay: high soluble salt concentration, swelling of clay mineral content, and porosimetry – i.e. texture

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[37] Rodriguez-Navarro et al., "The Role of Clays in the Decay of Ancient Egyptian Limestone Sculptures," *Journal of the American Institute for Conservation* 36, no. 2 (1997), p. 162.

[38] Rodriguez-Navarro et al., "The Role of Clays in the Decay of Ancient Egyptian Limestone Sculptures," *Journal of the American Institute for Conservation* 36, no. 2 (1997): p. 151-163; Arnold, "Nature of Reactions of Saline Minerals in Walls," *In Conservation of Stone II, Preprints of the International Symposium* (1981): p. 13-23; Price and Brimblecombe, "Preventing Salt Damage in Porous Materials," *Preprints of the Congress on Preventive Conservation Practice Theory and Research*, (London: International Institute for Conservation, 1994): p.90-93.

[39] C. Rodriguez-Navarro and E. Doehne, "Salt Weathering: Influence of Evaporation Rate, Supersaturation and Crystallization Pattern," *Earth Surface Processes and Landforms* 24, no. 3 (1999), p. 195.

and structure. Since petrographic characteristics cannot be altered, they will not be addressed in this paper. In order to mitigate the other two conditions, however, the use of surfactant has been explored. Following chapters investigate the influence of surfactants on the swelling properties of clays as well as review their role as desalination enhancers.

CHAPTER III – DESALINATION

3.1 Introduction

As already mentioned, salts are one of the main factors in the deterioration of the Ayyubid Wall stone as they are present throughout the wall. The most visible are in the form of crusts and powdery efflorescence. Removal of these salts, therefore, is a subject of great concern for the conservation of the wall. To date, salt crusts have been removed in the pilot project area using a hydro-mechanical system (JOS), where the amount of aggregate (sieved marble powder), air pressure, and water flow has been set to accommodate the characteristics of the masonry surface. In addition, concentrations of salt efflorescence in selected areas are to be removed by means of paper pulp poultices and dry mechanical cleaning.[40]

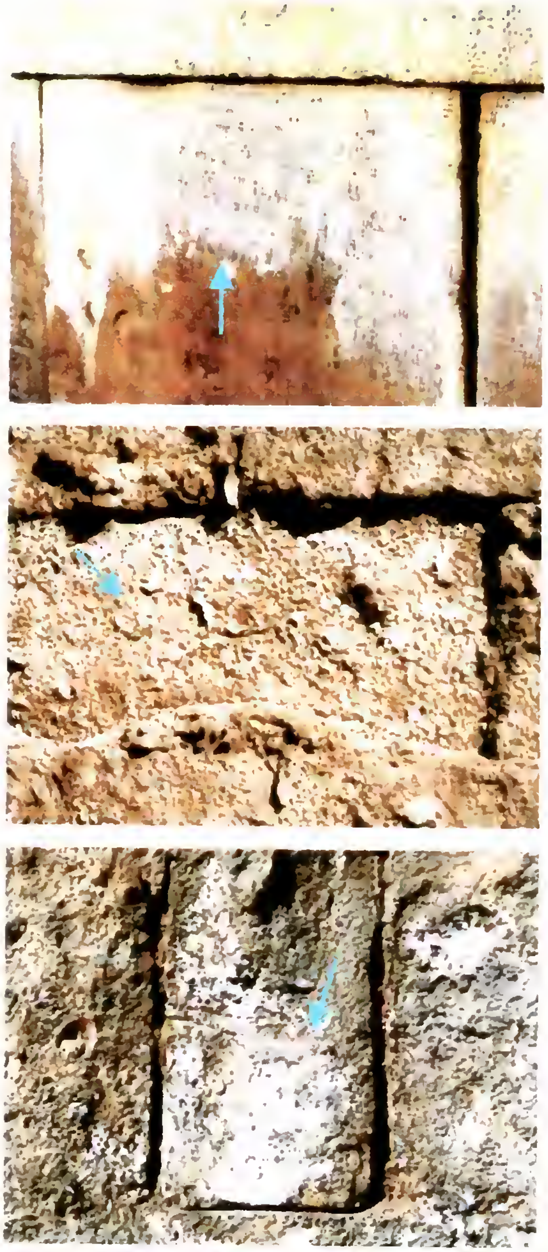

Fig. III.1 (Top) Thin salt crust on Comité replacement stone; (middle) salt veil and (bottom) salt bloom on Ayyubid stone. *From AKTC, 2001.*

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[40] The Aga Khan Trust for Culture, *The Eastern Ayyubid Wall of Cairo: Study Findings and Recommended Conservation Programme*. *Historic Cities Support Programs Technical Brief* no. 4 (2001), p. 59

Although mechanical removal of salt crusts is a necessary process, it only serves to clean the extant surface salts. It does not prevent future efflorescence since it does not address the source of the problem – i.e. the presence of mobile soluble salts. In contrast, poulticing is a technique commonly practiced in the field of conservation to extract water-soluble salts from inside the stone.

Poulticing is the application of a wet or damp inert absorbing support such as clay mixes or paper products onto the surface of the stone. When the moisture from the support penetrates the stone, salt crystals are dissolved. As the poultice dries, the salts migrate to the surface with the moisture front whereby they are absorbed into the support material.[41] With the removal of the poultice from the surface of the stone, salts accumulated in it are effectively removed.

While this technique has been successfully used in the past for the treatment of deteriorated Egyptian limestone sculptures, there has been some concern as to the effectiveness of the technique as the moisture from the poultice is unlikely to penetrate deep into the stone.[42] In fact, recent studies indicate that often times the poultices remove only the most superficial salts and that part of the dissolved salts can retreat deeper into the stone with the advancing moisture front. Although some salts are removed as the moisture migrates out into the poultice, many of the solvated ions move inside the stone

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[41] Nicholas Quayle, "Desalination of Stone by Partial Immersion and Poulticing," *Conservation News no.* 73 (United Kingdom Institute for Conservation of Historic and Artistic Works, 2000), p. 57.

[42] S. M. Bradley and S.B. Hanna, "The Effect of Soluble Salt Movements on the Conservation of an Egyptian Limestone Standing Figure," *Case Studies in the Conservation of Stone and Wall Paintings: In Preprints of the Contributions to the Bologna Congress* (London: International Institute for Conservation of Historic and Artistic Works, 1986), p. 58.

and re-crystallize when they return to or below the surface.[43] Also, there have been reported instances where the treatments first appeared successful but demonstrated active decay with extensive efflorescence in less than a decade.[44] Therefore, poulticing alone may not be effective enough, if at all.

As such, the use of a surfactant solution in the poultice may prove to be more effective in salt extraction as well as influencing subsequent salt crystallization. For this purpose, the role of surfactants in desalination and mitigation of salt decay is examined.

3.2 Surfactants (Surface Active Agents)

A surfactant, or a surface-active agent, is a term coined in the U.S in 1950, for detergents. In Europe, the term tenside (for tensio-active material) is more commonly used. For thousands of years, soaps were the only type of detergent used. However, their unstable character in hard or acidic water led to the development of alternatives in the early 20th century. Surfactants, including soaps, are essential components of a detergent, responsible for it its cleaning power.[45]

3.2 a - Surface Active Structure

A surfactant is a chemical compound that is capable of being preferentially adsorbed at a surface (gas, liquid or solid) or interface (liquid/solid, liquid/liquid, or gas/liquid) even at very low concentrations. That is, it changes the way the liquid, in

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[43] Nicholas Quayle, "Desalination of Stone by Partial Immersion and Poulticing," *Conservation News no.* 73 (United Kingdom Institute for Conservation of Historic and Artistic Works, 2000), p. 57.

[44] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 57.

[45] Ann Southall, "Detergents Soaps Surfactants," *Dirt and Pictures Separated* (The United Kingdom Institute for Conservation of Historic and Artistic Works, 1990), p. 29.

which it is dissolved, acts on that surface or interface. Such functional properties are due to the characteristic structure of the surface-active molecule. In order to better understand its working process, a simple soap (sodium laurate) is illustrated.

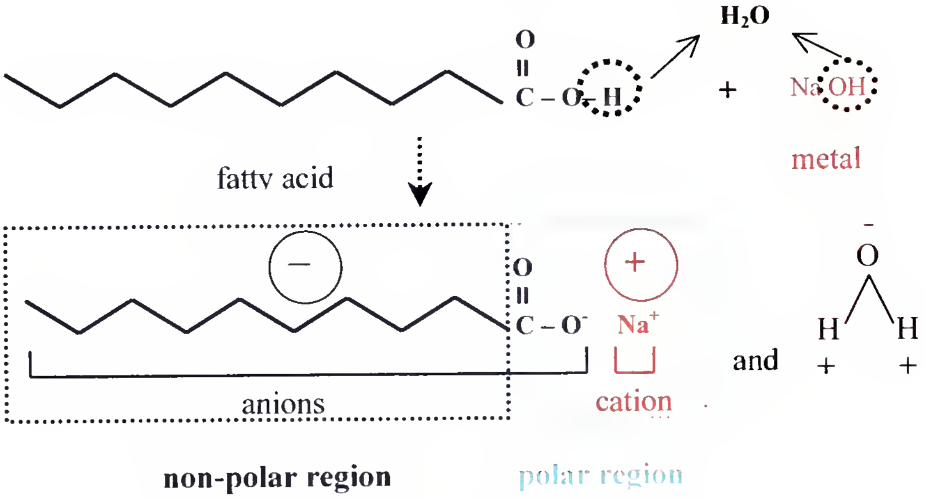

Fig. III.2 Formation of soap by ion dissociation.

Soap is formed by the reaction of a metal alkali with a carboxylic or fatty acid in water. When the molecules are dissolved in water, the OH^- from the alkali and H^+ from the acid dissociate, leaving the dissociated soap: sodium cations and laurate anions. The soap molecule, then, consists of two distinctive regions with different character and solubility properties: a hydrophilic (polar) head and a hydrophobic (non-polar) hydrocarbon tail. In fact, it is due to this amphipathic structure (having an affinity at both ends) that surfactants can be attracted to two immiscible solvents.

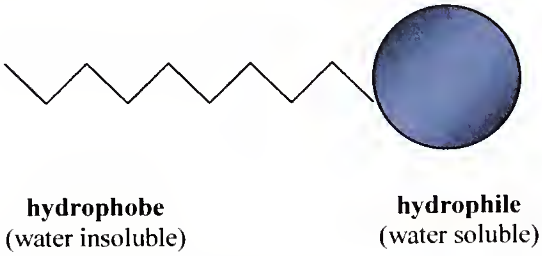

Fig. III.3 A schematic representation of a surfactant molecule

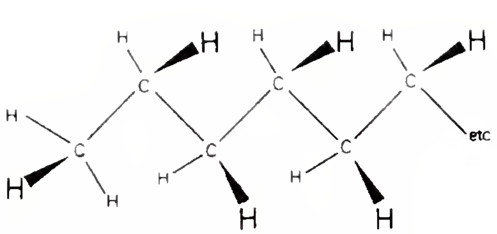

Fig. III.4 A structural representation of a hydrocarbon chain. *From Science for Conservators Vol. 2, 1992.*

The hydrophobic fatty chain is made of eight to eighteen hydrocarbons, which can be aliphatic, aromatic, or a mixture of both.[46] The hydrogens around the carbons form a tetrahedron. In most cases, the non-polar chain has an uneven number of carbon atoms with the final carbon forming the carboxyl (COOH) group (Figure III.1).[47] While this structure is similar in most detergents, the hydrocarbon chain may be derived from petroleum and other petrochemicals rather than from triglycerides or cholesterols.[48] Variations in the chain include branching, double bonds, and/or the presence of aromatic components.

The number of carbons and their structural features are significant to the way surfactants perform. For example, higher number of carbons with branching or multiple-chain hydrophobes increases the point of minimum surface tension and minimum critical micelle concentration, but can also lower the water solubility of the soap (see section *3.5* for details). Therefore, depending on the need, some surfactants will prove more effective than others.

3.2 b - Classes of Surfactants

Surfactants are categorised as ionic or non-ionic depending on their polar groups. Ionic surfactants can be classified either as cationic or anionic. Non-ionic surfactants may

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[46] Kiwi Web, "Surface Active Agents," [cited 28 October 2001]; available from URL: http://www.chemistry.co.nz/surfactants.htm

[47] The Crafts Council, *Science for Conservators Vol. 2: Cleaning* (New York: The Conservation Unit of the Museums & Galleries Commission and Routledge Publishers, 1992), p. 81.

[48] Ann Southall, "Detergents Soaps Surfactants," *Dirt and Pictures Separated* (London: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1990), p. 29.

have a polar end that is sufficient to ensure its solubility in water. Depending on the chemical structure and the nature of the solubilizing functionality, surfactants can be divided into four types: cationic, anionic, non-ionic, and amphoteric. This last type of surfactant is neither ionic nor non-ionic because it contains both positive and negative charges in the hydrophilic part with the ability to have more than one charge of either sign.[49]

Cationic surfactants work in acidic solutions and their hydrophilic group carries a positive charge. Their water solubility is given largely by amine and ammonium groups. Anionic surfactants have a negatively charged hydrophilic group such as carboxylates, sulphonates, sulphates and phosphates, and they work in neutral or alkaline solutions. In general, these two types of surfactants cannot be mixed. However, both are compatible with non-ionic and amphoteric surfactants. Non-ionic surfactants consist of a hydrophilic group that bears no charge in water and can be used at any pH. Water solubility is provided by highly polar ethylene oxide chains and hydroxyl groups, which readily form hydrogen bonds. Amphoteric surfactants have neutral molecules but contain both positive and negative charges in the active region, enabling them to function in acidic as well as alkaline solutions.

All surfactants are soluble in at least one phase of a liquid system. Although this is generally water, some are also or only soluble in hydrocarbons. It is important to note

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[49] D.C. Cullum, *Introduction to Surfactant Analysis* (New York: Blackie Academic & Professional, 1994), p. 17.

that while some surfactants may be insoluble in a non-polar solvent, they may still solubilize a non-polar material.[50]

3.2 c - Working Properties

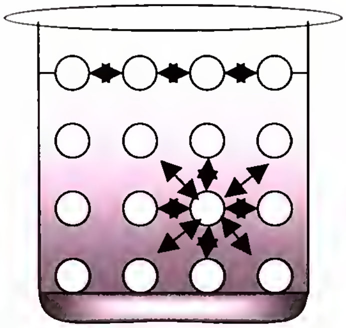

Fig. III.5 A schematic drawing of surface tension.

The role of a surfactant is to alter the surface activity of the liquid by reducing its surface tension. Such action is necessary due to the strong cohesive forces of liquid molecules. The cohesive force in a liquid pulls the neighboring molecules as close to one another as possible. For the molecules at the surface, this force is slightly altered because they do not have molecules on all sides. As a result, they exhibit stronger cohesions to the nearest neighboring surface molecules where the force applied across the surface is "like the skin pulled over a drum."[51] This effect of the intermolecular attractive forces at the surface, then, is referred to as surface tension.

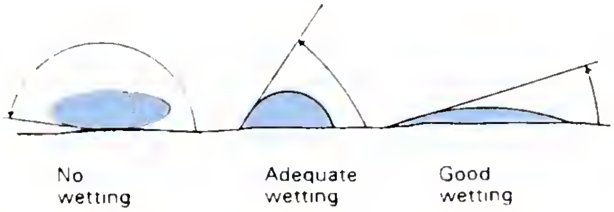

Fig. III.6 Water droplet and its contact angle. Due to the tendency of the liquid molecule to pull itself tight, it requires more energy for the droplet to increase its surface area. Water can flow without pressure and wet a surface when the contact angle is less than 90°. *From Dirt and Pictures Separated, 1990.*

When the liquid molecules are as close to one another as they can be, the liquid assumes a shape that has the smallest surface area for a given volume. For example, in a small

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[50] Ann Southall, "Detergents Soaps Surfactants," *Dirt and Pictures Separated* (London: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1990), p. 30.

[51] Kiwi Web, "Surface Active Agents," [cited 3 March 2002]; available from URL: http://www.chemistry.co.nz/propwat.htm.

quantity of liquid, this shape is a spherical drop (or droplet). A change in the shape increases the surface area, thereby requiring more energy to stretch the skin. Since every system tends toward a state of minimum energy, it is easier for a droplet to spread and wet the surface when the molecular cohesive forces are weak or if the skin tension is low.[52]

However, in the case of a solid surface, it cannot lower any excess surface energy by reducing the total interfacial area. In a solid-liquid interface, surface-active materials preferentially adsorb at the interface to reduce the imbalance of forces at the interface. In other words, the adsorption of molecules at a solid-liquid interface creates a transition region where the composition of the system changes from mainly that of the solid to mainly that of the liquid. Consequently, it is the ability of the surfactants to adsorb onto a solid surface from a solution that enables them to serve a multi functional use: they can be used to remove unwanted materials from a system (detergency), they can change the wetting characteristics of a surface (coating and waterproofing), or they can stabilize a finely divided solid system in a liquid (dispersion stabilization).[53]

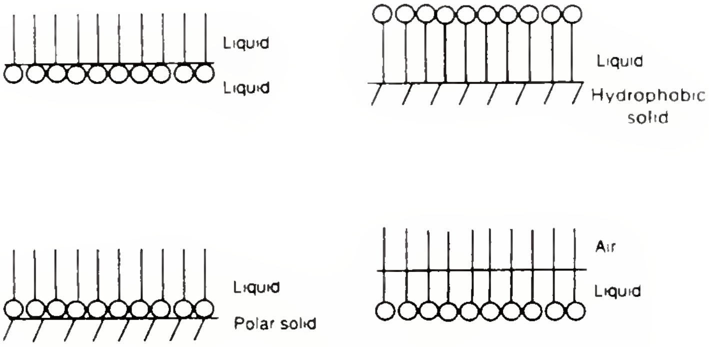

Fig. III.7 Adsorption at interfaces. *From M.R. Porter, 1994.*

There are numerous mechanisms by which a material may be adsorbed

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[52] G. Torraca, *Porous Building Materials: Materials Science for Architectural Conservation, 2nd ed.* (Rome: ICCROM, 1982), p. 9.

[53] Drew Myers, *Surfaces, Interfaces, and Colloids: Principles and Applications, 2nd ed.* (New York: Wiley-VCH, 1999), p.204.

onto a surface, but in general, the concentration of the surfactant molecules at the surface/interface is due to their polarity difference. However, the ability of the surfactants to produce a desired effect by adsorbing at the interface with a specific orientation depends on the natures of the solid, the surfactant, and the solvent.

For example, the surface tension of water is created by the strong cohesive force of water molecules. The attraction of water molecules to one another is greater than the mutual attraction to surfactant molecules or their attraction to water.[54] Therefore, the force holding the water molecules together pushes the surfactant molecules out to the boundaries preventing them from dispersing throughout the solution. As a result, the surfactant molecules concentrate at the surface and align themselves such that the non-polar end tries to escape the water while the polar end forms hydrogen bonds with the water molecules.[55] Since the cohesive force of the hydrocarbons is less than that of the water, they are held back by the polar groups and prevented from forming a separate phase. Instead, the surface of the water is coated with surfactant molecules resulting in the reduction of surface tension. As more surfactant is added to the water, the quantity of surfactant molecules at the surface increases, progressively lowering the surface tension. Eventually, there are a sufficient number of molecules on the surface to form a uniform hydrocarbon outer layer.

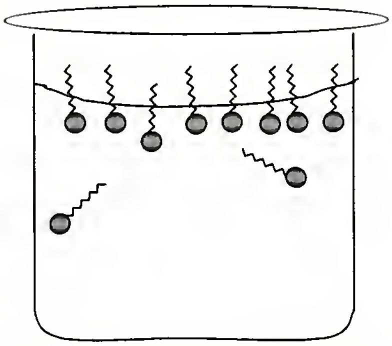

Fig. III.8 A schematic representation of the aligned surfactant molecules on the water surface.

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[54] Ann Southall, "Detergents Soaps Surfactants," *Dirt and Pictures Separated* (London: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1990), p. 30.

[55] Giorgio Torraca, *Solubility and Solvents for Conservation Problems, 4th ed.* (Rome: ICCROM, 1990), p. 44.

At equilibrium, or critical micelle concentration (CMC), the concentration of surfactant molecules is greater at the surface than within the body of the solution and the surface tension of the water remains constant. When this equilibrium is exceeded, small clusters of molecules or micelles form within the body of the solution and orient themselves such that the hydrophobic tails point toward the center and the polar ends point to the outer surface, forming a rough sphere. The micelles, then, increase the solubility rate of water-soluble materials with their polar ends. Water-insoluble materials such as grease or dirt, on the other hand, get suspended or solubilized in the center.[56]

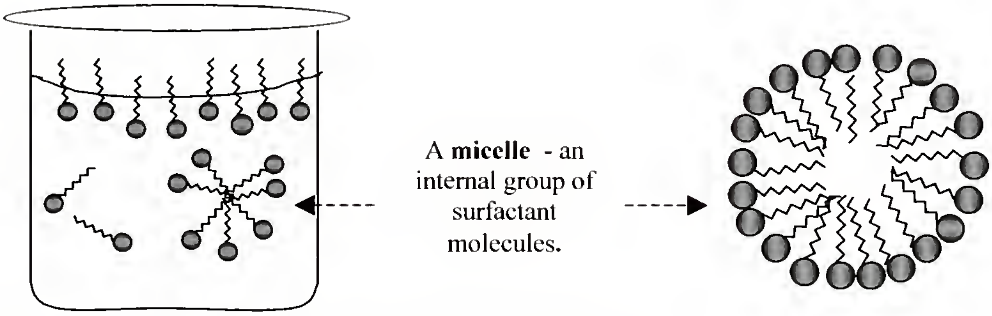

Fig. III.9 Surfactant molecules in a beaker of water. Adapted from http://www.chemistry.co.nz/propwat.htm and Giorgio Torraca, 1990.

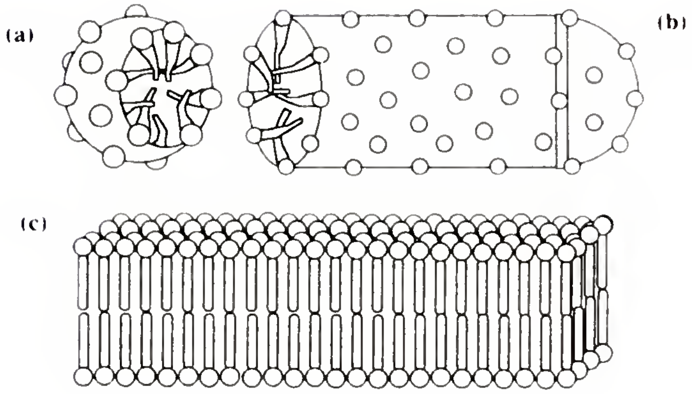

Fig. III.10 Shape of micelles. *From M.R. Porter, 1994.*

(a) Spherical
(b) rod-shaped or cylindrical
(c) lamellar

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[56] The Crafts Council, *Science for Conservators Vol. 2: Cleaning* (New York: The Conservation Unit of the Museums & Galleries Commission and Routledge Publishers, 1992), p. 83.

At or just above the critical micelle concentration, the micelles form a reservoir of molecules that keep the surfaces/interfaces saturated and the surface tension as low as possible. When more surfactant is added, the micelles keep their size, but grow in number. If the CMC is exceeded by ten or more times, an inversion occurs such that their number remains the same but their size and shape changes.[57] Therefore, the CMC point can be considered as the lowest concentration needed to get the maximum benefit of the surface-active properties.[58]

For the solid system, the mode and the extent of surfactant adsorption depend on the nature of the surface. Surfaces can be divided into three classes: those that are essentially nonpolar and hydrophobic, those that are polar but do not possess significant surface charge, and those that possess strongly charged surfaces.

The first type of adsorption occurs as a result of dispersion force interactions. Similar to the molecule orientation seen in water, the orientation of the adsorbed molecules will be such that the hydrophobic tail will be associated with the solid surface with the hydrophilic group directed toward the aqueous phase. Although the molecules initially lie along its interface, they gradually become perpendicularly oriented when they are at or near saturation, forming a "monolayer."[59]

Adsorption onto a polar, but uncharged surface is due to the potential forces such as the dispersion forces, dipolar interaction, and hydrogen bonding, operating at the surface. For this class, the relative balance between the dispersion forces and the polar

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[57] Ann Southall, "Detergents Soaps Surfactants," *Dirt and Pictures Separated* (London: The United Kingdom Institute for Conservation of Historic and Artistic Works, 1990), p. 31.

[58] M.R. Porter, *Handbook of Surfactants, 2nd ed.* (London: Blackie Academic & Professional, 1994), p. 36.

[59] Drew Myers, *Surfaces, Interfaces, and Colloids: Principles and Applications, 2nd ed.* (New York: Wiley-VCH, 1999), p. 205.

interaction determines the mode of adsorption: if the dispersion forces dominate, adsorption will occur in the same manner as the nonpolar surface. If the polar interactions dominate, adsorption occurs in a reverse mode where the surfactant molecules will orient themselves with the hydrophilic head toward the solid surface and the hydrophobic tail toward the aqueous phase.

For solids with a charged surface, which includes almost all inorganic oxides and salts, adsorption by all of the previously mentioned mechanisms is possible. The dominant mechanism, then, can go from ion exchange to ion bonding to dispersion or hydrophobic interactions. If adsorption occurs as a result of ion exchange where the adsorbed counterions are displaced by surfactant molecules, the electrical nature of the surface will not be greatly affected. In ion pairing, excess surfactant molecules are adsorbed and there is a net decrease in surface charge. Complete charge neutralization takes place as all of the accessible surface charges are paired with surfactant ions.[60] In dispersion or hydrophobic interactions, the particles will be stable and will not coagulate if repulsive

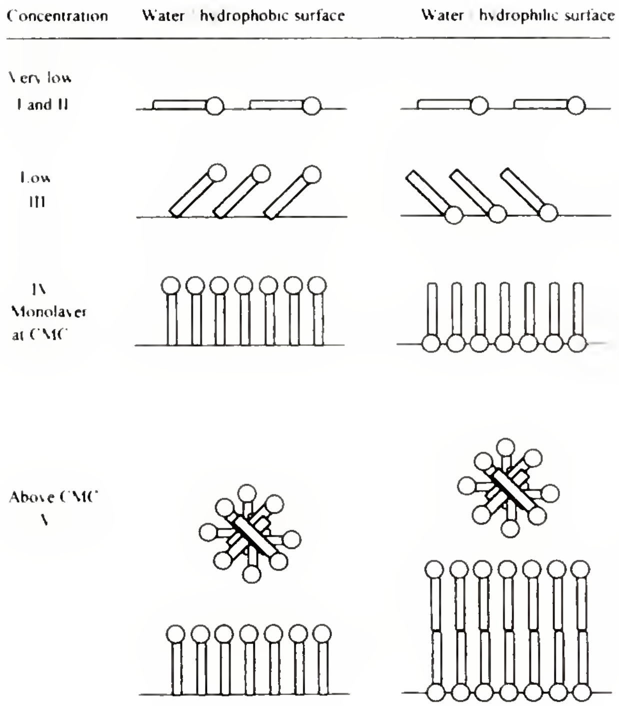

Fig. III.11 Adsorption and concentration. *From M.R. Porter, 1994.*

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[60] Drew Myers, *Surfaces, Interfaces, and Colloids: Principles and Applications, 2nd ed.* (New York: Wiley-VCH, 1999), p. 204 – 210.

forces between the particles can counter any attractive forces and there is a net repulsion between the attractive forces and the electrostatic repulsion.[61] It is important to note that an increase in electrolyte content generally causes a decrease in adsorption of surfactants onto surfaces of opposite charge and an increase in adsorption of like-charged molecules.[62] Therefore, ionic surfactants in the presence of electrolytes tend to reduce the electrostatic repulsion between the hydrophilic groups, increasing their tendency to form micelles. The critical micelle concentration of the surfactant, then, is significantly reduced when electrolytes are present in the solution.[63]

Surfactant adsorption by ion exchange or ion pairing results in the orientation of the molecules with their hydrophobic tail groups toward the aqueous phase. As such, the solid surface becomes hydrophobic and therefore difficult to wet. Moreover, once the solid surface has become hydrophobic, it is possible for adsorption to continue by dispersion force interactions. In such case, the charge on the surface will be reversed, acquiring a charge opposite to that of the original surface since the hydrophilic group will now be oriented toward the aqueous phase.[64]

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[61] M.R. Porter, *Handbook of Surfactants, 2nd ed.* (London: Blackie Academic & Professional, 1994), p. 82.

[62] Drew Myers, *Surfaces, Interfaces, and Colloids: Principles and Applications, 2nd ed.* (New York: Wiley-VCH, 1999), p. 204 – 210.

[63] M.R. Porter, p. 38.

[64] Myers, p. 210.

3.3 Potential Effects of Surfactants on Salt and Clay Deterioration

3.3 a - Surfactants and Salts

Due to their versatile adsorption capabilities, surfactants are utilized in various industries. Although their most notable applications are as cleaning agents, they have also been used in the field of conservation as biocides, surface-protection agents, clay-stabilizing agents, and stone desalination agents.[65] For the purpose of this paper, however, only the last two functions will be covered.

It has been affirmed that the mechanical stress arising from crystallization of soluble salts in pore walls is the primary destructive mechanism for stone deterioration.[66] There have been several proposals for the application of surfactants to mitigate salt damage since they have the ability to influence the physical properties of a salt solution such as its viscosity, surface tension, and vapor pressure. Consequently, surfactants can have a critical effect on the dynamics of solution flow and evaporation within the stone, thereby altering the degree of supersaturation, dynamics of precipitation, and salt growth.

In an early article, Pühringer and Engström theorized how surface-active substances could inhibit harmful salt structures by influencing their surface tension.[67] They argued that surfactants might reduce capillary transfer of salt solutions and subsequent salt precipitations by reducing the surface tension and increasing the viscosity of films of salt solutions. That is, if the wetability of the liquid is increased, it will result

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[65] Rodriguez-Navarro, Doehne, and Sebastian, "Influencing Crystallization Damage in Porous Materials through the Use of Surfactants: Experimental Results Using Sodium Dodecyl Sulfate and Cetyldimethylbenzylammonium Chloride," *Langmuir* 16, no. 3 (2000), p. 947.

[66] C. Selwitz and E. Doehne, "The Evaluation of Crystallization Modifiers for Controlling Salt Damage to Limestone," *Journal of Cultural Heritage 3,* (2002), p. 4.

[67] J. Pühringer and L. Engström, "Unconventional Methods for the Prevention of Salt Damage," *The 5th International Congress on Deterioration and Conservation of Stone* (1985): p. 241-250.

in thinning of the liquid film. This will in turn reduce the capillary transport capacity, preventing salts from migrating. Similarly, if the surfactants reduce the solution/stone contact angle, it results in faster capillary transport. In such a case, the changes in the flow of the saline solutions will induce the salts to crystallize on the stone surface rather than within the stone.

Another possible effect of surfactants may be on nucleation, the size of salt particles precipitated from saturated solutions that are dependent on the surface energy (supersaturation). Since salt crystallization (or re-crystallization) requires transfer of moisture to and from the surface of the salt formation, manipulation of the surface/interfacial tension in the water/salt solution and the substrate will alter the rate of hydration and dehydration of the salts, making it possible to control the growth rate and particle size of the precipitates. Alterations of salt's hydration behavior can also affect the strength of salt cement binder and its adhesion of salt crystals to the substrate. The agglomerations of salts depend on the accumulation of moisture in the pore system of the salt film and in the contact surface between the salt film and the substrate. Therefore, by modifying the amount of moisture present, the strength of salt cement can also be controlled. It is important to keep in mind, however, that certain combinations of surfactants with salts can just as well accelerate deterioration of materials. There is yet to be any detailed knowledge of application of surfactants for the control of processes involved in the formation of harmful salt.[68]

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[68] J. Pühringer and L. Engström, "Unconventional Methods for the Prevention of Salt Damage," *The 5th International Congress on Deterioration and Conservation of Stone* (1985): p. 241-250.

In 1990, Pühringer and Weber proposed an alternative approach to desalination.[69] This study was based on the use of surfactants to prevent salt damage by fostering the salt to "extract itself." It has been found that the formation of whiskers is one of the most effective extraction mechanisms. Therefore, if the crystals can be made to emerge as whisker efflorescence, they should enable further crystallization, thus depleting salt content in the material.

The mobility of liquid films on mineral surfaces is determined by the electrical charges on the boundary surface between the solid phase and the liquid, whereas the effect of a liquid transfer on material surfaces due to surface diffusion is determined by vapor pressure distribution above the surface of the salt solution film. The object of an extraction method, then, is to convert the bulk of liquid/salt bound to the material pores into thin mobile salt solution films capable of being transported through the pores and on material surfaces by making use of both vapor pressure and charge conditions in the boundary layer between liquids or materials which are, in theory, incompatible. In order to achieve this, there must be special boundary layers between the pore surfaces of the substrate material, the transferable salt solutions, and the extracting liquids where the charge conditions at the respective boundary surfaces are such that mobilization of salt solutions is facilitated or accelerated. The role of a surfactant, then, is to provide appropriate electrical charges on the material surfaces, whereby the surface active properties of a salt solution film can be altered, promoting separation of water and salt solution from the mineral substrates and inducing salt extraction. As such, the effect of a

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[69] J. Pühringer and J. Weber, "A Model for Salt Extraction – Some Principles." *Preprints of the 9th Triennial Meeting of the ICOM Committee for Conservation* (1990): p. 355-360.

surfactant depends on whether the surfactant pairing is to a charged surface of a substrate or to the surface of a fluid.

It has been shown that use of appropriate surfactants can influence the formation of structures and forces arising in the initial state of whisker growth in a favorable way. However, it is important to note that solutions containing different types of surfactants display different drying rates. Therefore, certain surface-active preparations influence the dynamics of salt structure, especially the whisker formation. Moreover, compared to anhydrates, hydrate salts are easier to mobilize and extract. The extraction effect, then, will differ between various salts and salt mixtures depending on their thermodynamics and the physico-chemical properties such as pore volume and geometry, chemical composition, and reactivity of the substrate.[70]

More recently, Rodriguez-Navarro, Doehne and Sebastian[71] studied the effects of both anionic and cationic surfactants on the process of salt crystallization in a porous calcareous stone in hopes to evaluate their use in desalination or mitigation treatments for stones affected by salt weathering. Observations revealed that preferential adsorption of the anionic surfactant on calcite surfaces significantly modifies the crystallization behavior of sodium sulfate, inducing crystallization and growth of non-equilibrium crystals, whereas in the presence of the cationic surfactant, the salt crystals form shapes that are closer to their equilibrium shape.

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[70] J. Pühringer and J. Weber, "A Model for Salt Extraction – Some Principles," *Preprints of the 9th Triennial Meeting of the ICOM Committee for Conservation* (1990): p. 355-360.

[71] Rodriguez-Navarro, Doehne, and Sebastian, "Influencing Crystallization Damage in Porous Materials through the Use of Surfactants: Experimental Results Using Sodium Dodecyl Sulfate and Cetyldimethylbenzylammonium Chloride," *Langmuir* 16, no. 3 (2000): p. 947-954.

Although it has already been established that surfactants induce kinetic and morphological changes in crystallizing salts, it appears that there is a direct relationship between the ability of the surfactant to form large amounts of micelles and the ability to induce nucleation of sodium sulfate at low supersaturation ratios. For example, due to its low adsorption onto calcite, the cationic surfactant concentrates in the bulk of the saline solution, producing rod-like or disk shaped micelles that have higher solubilization capacity than the spherical ones. These micelles, then, significantly enhance solute transport from the bulk solution toward growing crystal nuclei, resulting in crystallization at low supersaturation. The anionic surfactant, on the other hand, is less concentrated in the bulk saline solution due to its preferential adsorption onto calcite. This behavior is reflected in the limited number of micelles that change shape from sphere to rod or disk. Thus, both the amount of micelles and the solubilization capacity will be low, resulting in crystallization at high supersaturation.

Moreover, it has been found that since the cationic surfactant can induce early crystallization within stone pores at low supersaturation values, it will reduce crystallization pressure and cause less damage to the stone. By contrast, saline solution with the added anionic surfactant will reach a high supersaturation ratio before initiating crystallization, resulting in a high crystallization pressure and consequently more damage to the porous stone.

Differences in the solution transport can also be observed between the two types of surfactants: Saline solution in the presence of cationic surfactant displayed evaporation reduction, whereas the anionic surfactant seems to promote higher solution evaporation rate. However, such evaporation behavior is presumably due to salt crystallization within

the stone pore system (filling the pores with salts) induced by both types of surfactants rather than as a product of the surfactants' ability to change the contact angle, surface tension, and viscosity of the saline solutions.

While the greater evaporation rate of sodium sulfate with the anionic surfactant results in faster transport toward the evaporation front located just below the stone surface, allowing the salts to crystallize as harmful subflorescence, the cationic surfactant has greater tendency to promote even crystallization throughout the stone pores which may eventually deteriorate the stone if the dehydrated crystals re-hydrate. Such disadvantages imply that although differences in surfactant adsorptions and micelle shape evolution significantly modify salt crystallization behavior, the two types of surfactants tested are not an effective method for stone desalination when salts with hydrates are present. Therefore, additional research is necessary where effects of other surfactants or surfactant mixtures are evaluated.[72]

3.3 b - Surfactants and Clays

Another possible surfactant application is to reduce stone deterioration by mitigating the swelling of clays. According to Wendler, Klemm, and Snethlage,[73] the swelling behavior of clay minerals is due to the ability of the cations, which are fixed at the negatively charged centers of clay mineral layers, to form hydration shells. The size of the shells depends on the type of cation and the amount of water available. Therefore,

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[72] Rodriguez-Navarro, Doehne, and Sebastian. "Influencing Crystallization Damage in Porous Materials through the Use of Surfactants: Experimental Results Using Sodium Dodecyl Sulfate and Cetyldimethylbenzylammonium Chloride." *Langmuir* 16, no. 3 (2000): p. 947-954.

[73] Wendler, Klemm, and Snethlage, "Consolidation and Hydrophobic Treatment of Natural Stone," *Durability of Building Materials and Components. Proceedings of the 5th International Conference* (New York: E. & F.N. Spon. 1990): p. 203-212.

in order to prevent or reduce the hydration shell formations, it is necessary to block the negatively charged centers of the clay layers.

Earlier investigations have demonstrated that cations between clay layers such as Na$^+$, Ca$^+$, Mg$^+$, and K$^+$, are partially exchangeable with alkyl-ammonium ions. The extent of ion exchange depends on the location and the type of cation. Therefore, an experiment was carried out utilizing bifunctional alkyl-α-ω-diammonium chlorides on clay-rich sandstone samples. The goal was to replace the binding cations at the mineral interfaces with surfactant ions. Since both functional groups can be fixed at the negatively charged surfaces of clay layers, it was believed that this type of surfactant could achieve a stable configuration. Comparisons in results showed that among the alkyl diammonium ions with carbon chains ranging from C_2 to C_{12}, diammonium chloride with C_4 chains proved to reduce the hygric dilatation the most.[74] In fact, the reduction was about half of the original value.[75] The effect of the

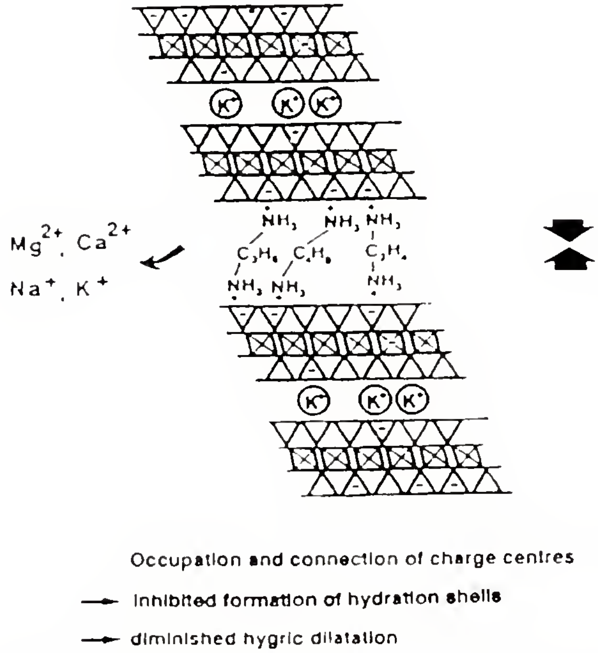

Fig. III.12 Model of the ionic exchange of cations against bifunctional cationic surfactants on clay mineral basal planes. *From Wendler, Klemm, and Snethlage, 1990.*

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[74] Snethlage and Wendler, "Surfactants and Adherent Silicon Resins – New Protective Agents for Natural Stone," *Material Research Society Symposium Proceedings* 185 (1991), p. 194.

[75] Wendler, Klemm, and Snethlage, "Consolidation and Hydrophobic Treatment of Natural Stone," *Durability of Building Materials and Components. Proceedings of the 5th International Conference* (New York: E. & F.N. Spon, 1990), p. 203-212.

butyl diammonium ions, then, is "to block the micropores within the clay mineral structures which are responsible for the swelling."[76]

The use of surfactants as clay-stabilizing agents can also be seen in studies performed by Wendler, Charola, and Fitzner. Due to the severe hygric swelling observed in the Easter Island tuff, the bifunctional surfactant BDAC (butyl di-ammonium chloride) was tested. This type of surfactant has been known to reduce swelling of various materials without affecting their hydric transport properties. Again, the idea was to allow preferential bonding between the surfactant and the reactive mineral surfaces, thereby preventing their hydration in the presence of water. Their results indicate that treatment with the anti-swelling surfactant significantly reduces both the hygric and hydric dilatation.[77]

Evaluation of the surfactant BDAC in mitigating the expansive properties of clays in the presence of salts has also been performed at the University of Pennsylvania.[78] E. Bourguignon studied the possible effects that BDAC had on two types (nummulitic and non-nummulitic) of Egyptian limestone, both containing a high soluble salt content (approx. 6% - 7% w/w), but not a significant amount of clay (acid insoluble residue approx. 3% w/w).

According to the author, the dimensional change experienced by the samples when subjected to wet/dry cycling was not greatly affected by application of the

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[76] Snethlage and Wendler, "Surfactants and Adherent Silicon Resins – New Protective Agents for Natural Stone," *Material Research Society Symposium Proceedings* 185 (1991), p. 195.

77 Wendler, Charola, and Fitzner, "Easter Island Tuff: Laboratory Studies for its Consolidation," *Proceedings of the 8th International Congress on Deterioration and Conservation of Stone* (Berlin: Möller Druck und Verlag, 1996), p. 1161.

[78] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 114.

surfactant, as all samples displayed a minimal cyclic variation. This is most likely due to the low clay content found in all samples. However, depending on the texture and the porosity of the stone, the surfactant seemed to reduce the amount of moisture absorbed by the sample during the wet/dry cycling. Moreover, the extent of weight gain (as a result of moisture absorption) seemed to depend on the presence of salts. That is, for the samples that were impregnated with salt, the surfactant proved to reduce the amplitude of weight difference during the wet/dry cycling. For those without salt-impregnation, an increase in the amplitude of weight difference was observed. Also, the application of surfactant reduced powdering of salt-impregnated stones, especially for the non-nummulitic type.[79]

Another important observation made by Bourguignon was the alteration in halite crystal formation as a result of the surfactant. Scanning Electron Microscopy (SEM) revealed that the presence of the surfactant within the stone resulted in the formation of much smaller NaCl crystals.[80] Therefore, M. McCormack further explored the use of the surfactant in controlling salt crystallization by examining the effects the surfactant had on desalination.

Her results showed that when porous limestone with high clay content (acid-insoluble residue between 30-40% w/w*) was poulticed, the samples treated with BDAC showed an increase in the desalination effectiveness by about 33-66%.[81]

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[79] E. Bourguignon, *Study of Deterioration Mechanisms and Protective Treatments for the Egyptian Limestone of the Ayyubid City Wall of Cairo* (master's thesis, University of Pennsylvania, 2000), p. 157.

[80] M. McCormack, *Conservation Studies for the Ayyubid City Wall, Cairo* (master's thesis, University of Pennsylvania, 2001), p. 46.

* Soluble salt content unknown

[81] M. McCormack, p. 54.

CHAPTER IV – METHODOLOGY & RESULTS

4.1 Treatment Overview

The goal of this research was to determine if either or both types of surfactants, the non-ionic Triton™ XL-80N and the anionic Orvus WA paste, would serve as desalination enhancers for the Ayyubid City Wall when applied using the poulticing technique. Therefore, tests were performed on the Egyptian limestone samples that are of the same type as the proposed replacement stone for the wall. In order to assess the possible influence of the surfactants, the samples were first impregnated with sodium chloride and subjected to wet-dry cycles. Once prepared, they were poulticed with different combinations of water and surfactant solutions where the amount of salt extracted from the stone was quantified by conductimetric measurements and NaCl titrator strips.

4.2 Sample Limestone Properties

While the mechanical and the physical characteristics of the representative replacement stone sample have been characterized by the Rock Engineering Laboratory of Cairo University (Table IV.1), its chemical analysis has been performed at the University of Pennsylvania. Two limestone cubes were taken and crushed in an agate mortar to obtain a uniform coarse powder. Using the Balance Denver Instrument XE-510, 50.00 (g) of the ground sample from each cube were measured and placed in a beaker with 500 (mL) of deionized water. The beaker was then placed on a magnetic stirrer with a magnetic stirring bar for 2 hours. The suspension was left to settle overnight and

filtered the following day. The solution obtained was put aside for salt identification discussed below. The solid left in the filter paper was weighed and put to dry in a Fisher Scientific Isotemp 500 series oven at 100°C for 24 hours and left to cool to room temperature in a desiccator supplied with silica prior to being weighed again. This procedure was repeated until the difference in weight of the two consecutive weighings was less than 0.01% of the weight of the sample (Appendix A). Here is how the weight of the soluble salt content for each sample was calculated:

Soluble salt % w/w = [(wt. of original sample – wt. of salt-free sample)] • 100 / wt. of original sample

therefore,

$$\text{Soluble salt \% w/w} = [(50.00\ \text{g} - 47.63\ \text{g})] \cdot 100 / 50.00\ \text{g}$$
$$= 4.74\ \%$$

Once the soluble salt content was quantified, the dried solid was placed in a beaker with 50 (mL) of deionized water to determine the amount of acid insoluble material present. This was achieved by adding HCl (14%) to the solution over a 24-hour period. When the solution ceased to effervesce in the presence of HCl indicating that all calcium carbonate ($CaCO_3$) had been dissolved, the solution was filtered. The filtered material consisted only of finely textured impurities (i.e. clays) without large mineral particles. The fines were dried in an oven at 100°C for 24 hours and left to cool to room temperature in a desiccator before being weighed again. This procedure was repeated

until a constant weight was achieved (Appendix B). The amount of fines can be expressed as a % w/w of the sample[82]:

% fines = (weight of dry fines / weight of original sample) • 100

therefore,

% fines (acid insoluble residue) = [5.1 (g) / 50.00 (g)] • 100

= 10.2 %

Table IV.1 *From the Rock Engineering Laboratory of Cairo University.*

Mechanical Properties

Uniaxial Compressive Resistance (kg/cm^2)	*Young's Modulus (kg/cm^2)*	*Tensile Resistance (kg/cm^2)*	*Double Shear Resistance (kg/cm^2)*
206.8	N/A	21	N/A

Physical Properties

Density (g/cm^3)	*Porosity (% by volume)*	*Water Absorption (%)*	*Main Mineral Composition (XRD)*
1.99 (average)	19.27 (average)	8.49 (average)	Calcite with small amounts of dolomite, quartz, halite and clay minerals

Table IV.2 Soluble salt identification.

Chemical Analysis

Total Soluble Salts (Average % w/w)	*Acid Insoluble Residue (Average % w/w)*	*Chloride (Cl^-)*	*Sulphate (SO_4^{2-})*	*Nitrate (NO_3^-)*	*Nitrite (NO_2^-)*
4.68 ± 0.06	9.28 ± 0.92	15 (mg/g)	> 16 (mg/g)	0.5 (mg/g)	0.00

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[82] J.M. Teutonico, *A Laboratory Manual for Architectural Conservators* (Rome: ICCROM, 1988), p.116.

With the obtained filtered solution that was put aside from the first step, a semiquantative analysis of the soluble salts present in the sample was determined using Merckoquant® analytical test strips. In order to simplify calculations, an additional 50 (mL) of deionized water was added to the solution to bring the final volume to 500 (mL). Aliquots were poured into small beakers and tested for various ions and their concentrations (Table IV.2). If the concentration obtained in a sample was at the maximum value of the indicators, it was necessary to dilute the aliquot to a known volume. Since the concentration of the ions in the solution is given in (mg/L), the concentrations of individual types of ions in the original sample were calculated as follows:

Ion concentration (mg/g sample) = x (mg/L) • vol. Solution (mL) • 1/1000 (L/mL) / wt. of original sample (g)

As for the diluted aliquot, the following factor was added to the above formula:

Diluted vol. (mL) / aliquot measured (mL)

therefore,

Chloride ion concentration (mg/g) = 1500 (mg/L) • 500 (mL) • (1/1000) (L/mL) / 50.00 (g)

Chloride ion concentration = 15 (mg/g)

The results obtained reflect the accuracy of the values when compared with the theoretical soluble salt content. That is, when the individual ion concentrations are calculated proportionate to its corresponding salt type, the sum of all soluble salt content theoretically present is close in value to the sum of the soluble salt content measured in the laboratory (Table IV. 3). For example, based on the given information, one can calculate the weight of NaCl present where:

Given: Molecular Weight of Cl^- = 35.45 (g) and NaCl = 58.44 (g)
Wt. of Cl^- obtained from the analytical test strip = 15 (mg/g)

then,

M.W. of salt type (g) / M.W. of corresponding concentrated ions (g) • Wt. of ion concentration (mg/g) = Weight of salt type concentration

therefore,

58.44 (g) NaCl / 35.45 (g) Cl^- • 15 (mg/g) Cl^- = 24.73 (mg/g) NaCl

When the theoretical weights of the four types of salts (Cl^-, SO_4^{2-}, NO_3^-, NO_2^-) are added, the sum of the calculated weights is 52.29 (mg/g). This is multiplied by the weight of the initial sample:

52.29 (mg/g) • 50.00 (g) = 2614.5 (mg)
2614.5 (mg) / 1000 = 2.6145 (g)

However, since the obtained values for the soluble salt contents were calculated as a percentage of w/w, the theoretical value of 2.615 (g) of soluble salts found in 50.00 (g) must be converted to a percentage by weight. Therefore,

2.615 (g) / 50 (g) • 100 = 5.23 % (g salt /g sample)

The theoretical value of 5.23% correlates closely with the laboratory obtained average value of 4.68 ± 0.06 % (Table IV.2).

Table IV. 3 Calculations of theoretical soluble salt content based on the obtained values.

	M.W. (g)	*Obtained wt. (mg/g)*	*Calculated wt. (mg/g)*	*Weight of salt per 50 (g) sample (g)*	*Total Salt (% w/w)*
Cl^-	35.45	15	---	---	---
Sodium Chloride ($NaCl$)	58.44	---	24.72	1.24	2.47
SO_4^{2-}	96.06	< 16*	----	---	---
Calcium Sulfate Dihydrate- a.k.a. Gypsum ($CaSO_4 \cdot 2H_2O$)	172.17	----	26.88	8.61	2.68
NO_3^-	62.00	0.5	---	---	---
Sodium Nitrate ($NaNO_3$)	84.99	---	0.69	4.25	0.069
Sum of Salts	---	---	52.29	---	5.22

* Since 15 mg/g is the highest value possible according to the indicator strip, this value was used for all calculations.

4.3 Experimental Samples and Materials

Fig. IV.1 36 limestone cubes from Cairo, Egypt.

Thirty-six pre-cut Egyptian limestone samples, each with an approximate dimension of 5x5x5 cm, were brought to the Architectural Conservation Laboratory at the University of Pennsylvania. Using a permanent marker (Sharpie®), each sample was labeled 1 to 36 at the upper left corner and weighed. The cubed samples were placed in an oven at 60°C for 48 hrs. After 48 hours, the cubes were taken out of the oven and allowed to cool in a dessicator for 12 hours prior to being weighed again. Following the initial 48-hour oven-drying procedure, a cycle of 24-hour oven drying and 12-hour

cooling in the desiccator was repeated until the difference between two successive weighings at 24-hour intervals was equal to or less than 0.1% of the weight of the sample (Appendix C). Once a constant weight was attained, the cubes were divided into 12 groups (each group consisting of 3 samples) where each group was prepared and/or subjected to different conditions and treatments. Identical treatments were performed for all three samples in each group.[83]

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[83] Adapted from NORMAL 11/85.

4.3 a – Salt

Several groups of the limestone cubes were impregnated with a saturated solution of sodium chloride (Fisher Scientific Company). Listed below are the main properties.[84]

Table IV.4 Physical properties of NaCl. *From CRC Handbook of Chemistry and Physics, 1994.*

<table>
<tr><th colspan="30">NaCl (Halite)</th></tr>
<tr><td colspan="5"><i>Molecular Weight</i></td><td colspan="5"><i>Density or Specific Gravity</i></td><td colspan="5"><i>Melting Point (℃)</i></td><td colspan="5"><i>Boiling Point (℃)</i></td><td colspan="5"><i>Solubility in cold water (0℃)</i></td><td colspan="5"><i>Solubility in hot water (100℃)</i></td></tr>
<tr><td colspan="5">58.44 g mol^{-1}</td><td colspan="5">$2.165^{25}{}_{4}$</td><td colspan="5">801</td><td colspan="5">1413</td><td colspan="5">35.7 g per 100cc</td><td colspan="5">39.12 g per 100cc</td></tr>
<tr><th colspan="30">Saturated NaCl solution at 20°C</th></tr>
<tr><td colspan="6"><i>Concentration</i></td><td colspan="6"><i>Density</i></td><td colspan="6"><i>Surface Tension</i></td><td colspan="6"><i>Viscosity</i></td><td colspan="6"><i>Vapor Pressure</i></td></tr>
<tr><td colspan="6">26.41% per weight</td><td colspan="6">1.198 g cm^{-1}</td><td colspan="6">8.35 Pa</td><td colspan="6">1.986 mPoise</td><td colspan="6">1.7634 kPa</td></tr>
</table>

4.3 b - Surfactants

The two types of surfactants chosen for the experiment were the non-ionic Triton™ XL-80N surfactant manufactured by Union Carbide Corp. and an anionic Orvus WA paste surfactant manufactured by Procter & Gamble. These two were considered preferable because neither of them contained chlorides. Since the desalination experiment entailed the impregnation of NaCl, it was believed that more accurate results could be assessed if there were no additional chlorides from the surfactants interfering with the final results. Moreover, these two surfactants proved to be more economical than the previously tested BDAC. Listed below are the main properties for the chosen materials.

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[84] David R. Lide, ed., *CRC Handbook of Chemistry and Physics* (London: CRC Press, Inc., 1994), p. 4-98

Table IV.5 Material Data for Triton™ XL-80N and Orvus WA paste surfactants.

Company Name	Union Carbide Corp.	Procter & Gamble
Product Name	**Triton™ XL-80N**	**Orvus WA paste**
Chemical Name	Alkyloxypolyethyleneoxypoly - propyleneoxy ethanol	N/A
Chemical Family	Alcohol Alkoxylate	N/A
Formula	$R\text{-}O\ (CH_2CH_2O)\ n\ (CH_2CHCH_3O)\ nCH_2\ CH_2OH$	N/A
Ingredients	N/A	Water, sodium alkyl sulfate and minor ingredients
Molecular Weight	420 g/mol (approx.)	N/A
Solubility in Water	Complete	Complete
Flash Point (Pensky-Martens Closed Cup ASTM D93)	260°F	> 200°F
Percent Volatile (% by weight)	0.56	N/A
Boiling Point (°F)	Decomposes at 760 mmHg	> 210°F
Freezing Point (°F)	27°F	N/A
Specific Gravity (H_2O = 1)	0.985	1.04
Vapor Pressure (mmHg)	< 0.01	20
Vapor Density (air = 1)	> 10	N/A
Evaporation rate (Butyl Acetate = 1)	< 0.01	N/A
Stability	Stable	Stable
Appearance	Murky liquid	White paste with faint detergent odor
Toxicity	N/A	Non-toxic
Cost	$11.25 (pint)	$5.75 (pint)

4.4 Methodology

4.4 a - Capillary Absorption Rate, Total Immersion, and Drying Curve

Following the test standards found in NORMAL 11/85, Group A (sample nos. 1, 2, and 3) was subjected to the capillary water absorption rate test. This was performed in order to determine the amount of water absorbed by the representative samples per unit surface area as a function of time at room temperature and pressure (Graph IV.1). The equation is as follows:

$$M_i = [m_i - m_0] / S$$

where, m_i = weight (g) of the sample at time t_i,

m_0 = weight (g) of the sample at time t_0,

S = surface of the sample in contact with water (cm^2)

In order to obtain uniformity, the three samples were placed in a plastic container above glass rods. The purpose of the glass rods was to reduce the resting areas of the samples. Throughout the capillary rise experiment, the samples were placed so as to avoid coming into contact with one another, but the bottoms of the cubes were in contact with deionized water at a level just above the glass rods. The container, then, was closed with a tight lid to avoid evaporation, but not hermetically sealed since the latter can cause condensation and possibly wet the samples on other surfaces. At given intervals, the samples were taken out of the container, patted-dry using a damp paper towel, weighed, and placed immediately back in the container. This process was repeated until the difference in the amount of absorbed water in two successive weighings at a 24-hour interval was equal to or less than 0.1% of the amount of total water absorbed (Appendix

D). At this point, the asymptotical value *(M*)* of the capillary absorption curve was considered reached and calculated as follows:

$$M^* = M_i \text{ where, } [(M_i - M_{i-1}) / M_i] \bullet 100 \leq 0.1\%$$

where, M^* = asymptotical value of the amount of absorbed water per unit surface of the sample (g/cm^2),

M_i and M_{i-1} = amount of water absorbed per unit surface of the sample at times t_i and t_{i-1} (g/cm^2)

With the capillary absorption rate determined, the samples were immersed in deionized water for 24 hours to insure complete saturation (NORMAL 7/81). The amount of water absorbed by the samples when completely immersed in water at room temperature and pressure can be expressed as a percentage of the dry weight of the samples and the values can be equated to the % Apparent Porosity (ASTM C948-94). The amount of water absorbed *(ΔM/M %)* by the sample at time t_i is calculated as such (Graph IV.2):

$$\Delta M/M\ \% = [(M_i - M_0) / M_0] \bullet 100$$

where, M_i = weight (g) of the sample imbibed with water at time t_i,

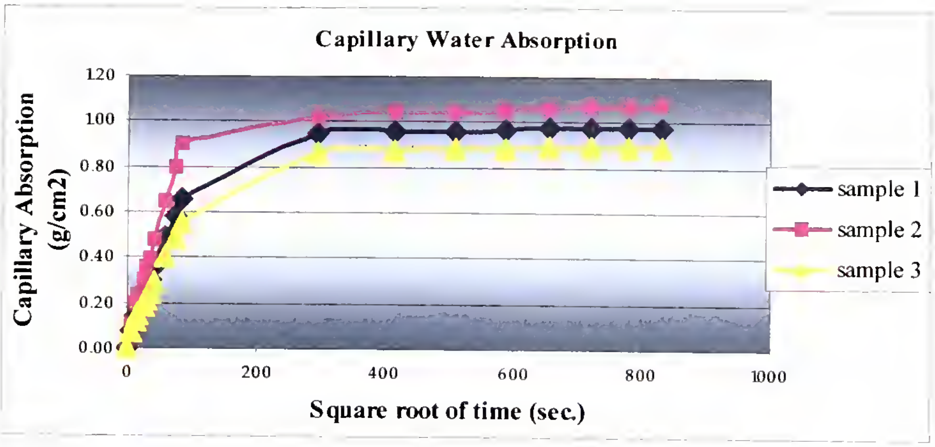
M_0 = weight (g) of the dry sample

Following the 24-hour total immersion, the samples were monitored to assess their drying behavior (NORMAL 29/88). That is, the loss of weight by evaporation of water over time was measured and expressed as a percentage of the dry weight of the sample. Such measurements allow one to calculate the hypothetical time required for the loss of water content to reach the asymptotical value (Appendix E).

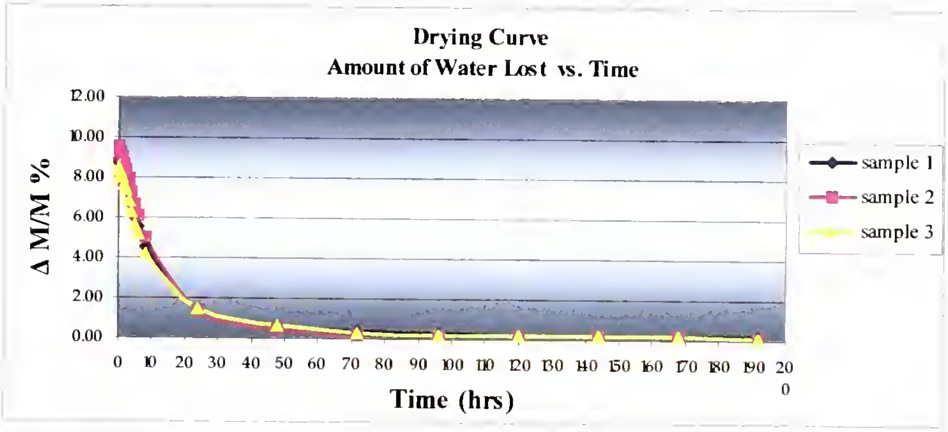
The three samples were taken out of the container and patted-dry using a damp paper towel. They were then weighed and placed on a metal rack to provide proper air circulation. The metal rack was placed in an open environment to allow the samples to air-dry in a uniform fashion. The room temperature varied from 22°C to 25°C and the relative humidity was between 31% ~ 40%. Again, the sample weights were measured at given intervals until the asymptote was obtained. The differences in relative moisture content at different times for the samples are represented as a function of moisture content in Graph IV.3 (See Appendices D and E for all pertinent calculations.)

Once the experimental values were obtained, the samples were dried in an oven at approximately 60°C until constant weight was achieved. Here, the weight is considered constant when the difference between two weighings taken at a 24-hour interval is equal to or less than 0.01% of the weight of the dry sample (Appendix E).

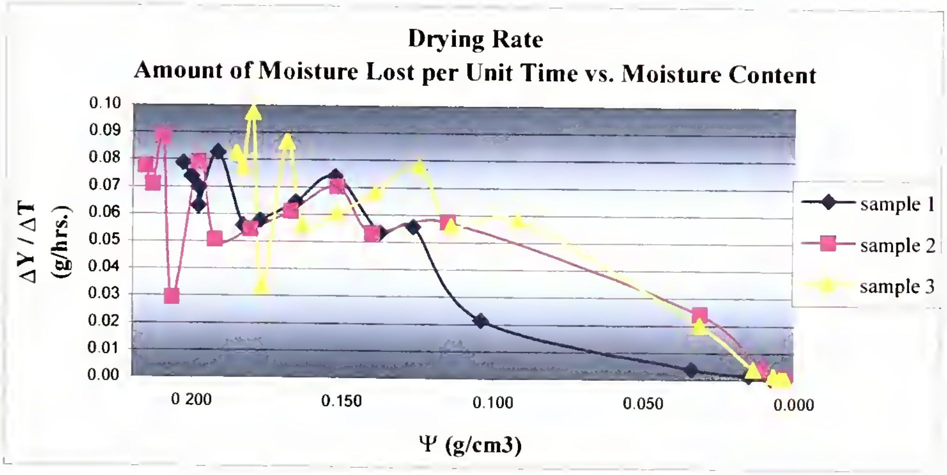
Graph IV.1 Capillary Water Absorption.



Graph IV.2 Drying Behavior after Total Immersion.



Graph IV.3 Drying Rate.



4.4 b - Salt Impregnation

Fig. IV.2 Samples immersed in NaCl bath.

Since the solubility of NaCl in water (0°C) is 35.7 (g) per 100 (mL), a total of 6.48 (kg) of NaCl crystals were added to 17.5 (L) of deionized water to assure saturation. The saline solution was prepared in a beaker over a magnetic stirrer. When the NaCl crystals were visibly floating in the solution, it was considered saturated. The solution was poured into a large plastic container where the remaining nine groups of cubes, numbered 10 to 36, were carefully placed above glass rods so as to allow penetration on all sides of the cubes.

The samples were immersed in the solution for 24 hours. The cubes were then removed from the solution and left to air-dry for 10 hours in room temperature and humidity. This method of drying results in fewer hairlines cracks and visibly reduces the appearance of efflorescence compared to placing the samples immediately in the oven to dry.[85] The number of hours, however, was determined by the Drying Curve (Graph IV.1) which indicated 10 hours as the time needed to reach the critical moisture content in the limestone samples.

Following the 10-hour air-drying, the samples were weighed and dried in an oven at approximately 105°C for 24 hours and left to cool to room temperature in a desiccator before being weighed again. This procedure was repeated until a constant weight was

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[85] Charola, Nunberg and Freedland, "Salts in Ceramic Bodies I: Introducing Salts into Ceramics," *International Journal for Restoration of Buildings and Monuments* 7, no. 2 (2001), p. 123.

attained (Appendix F). The weight of salt introduced into the cubes was calculated as follows:

wt. of salt introduced (g) = wt. of salt-deposited sample (g) - wt. of initial sample (g)

or

wt. of salt introduced (% w/w) = [wt. of salt-deposited sample (g) - wt. of initial sample (g) / wt. of initial sample (g)] • 100

Fig. IV.3 27 samples impregnated with NaCl.

Fig. IV.4 A close-up view.

Table IV.6 Amount of NaCl introduced.

	A	C	D	E	F	
sample no.	initial wt. of sample (g)	wt. of sample after NaCl impregnation (g)	wt. of NaCl introduced (g) [C-A]	amount of NaCl per stone sample (% w/w) [(D/A) *100]	average amount of NaCl introduced (%w/w)	stdv. (±)
4 5 6	*control samples - no salt introduced*					
7 8 9	*control samples - no salt introduced*					
1 2 3	*control samples - no salt introduced*					
16	299.66	304.00	4.34	1.45	1.72	0.22
17	282.52	287.38	4.86	1.72		
18	285.58	291.27	5.69	1.99		
19	301.33	300.54	-0.79	-0.26	1.56	0.08
20	283.90	288.54	4.64	1.63		
21	295.68	300.06	4.38	1.48		
28	291.63	295.79	4.16	1.43	1.35	0.11
29	285.46	288.86	3.40	1.19		
30	296.84	301.07	4.23	1.43		
13	279.61	284.00	4.39	1.57	1.41	0.11
14	289.38	293.27	3.89	1.34		
15	287.22	290.99	3.77	1.31		
22	318.26	323.32	5.06	1.59	1.50	0.15
23	294.10	297.91	3.81	1.30		
24	293.57	298.34	4.77	1.62		
25	286.72	290.96	4.24	1.48	1.55	0.06
26	278.00	282.52	4.52	1.63		
27	287.38	291.85	4.47	1.56		
10	280.56	284.94	4.38	1.56	1.52	0.17
11	296.95	301.99	5.04	1.70		
12	292.98	296.75	3.77	1.29		
34	292.39	296.79	4.40	1.50	1.65	0.12
35	286.47	291.18	4.71	1.64		
36	278.12	283.13	5.01	1.80		
31	287.98	292.23	4.25	1.48	1.64	0.20
32	279.93	285.32	5.39	1.93		
33	295.49	299.96	4.47	1.51		

4.4 c - Wet / Dry Cycles

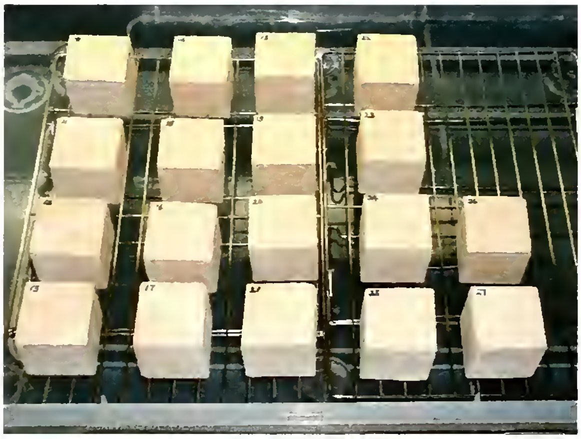


Fig. IV.5 27 cubes subjected to wet cycles.

Once the 27 sample cubes were completely dried, each sample was brushed on all sides to remove any efflorescence present. The samples were then subjected to wet-dry cycling to simulate the damaging environmental conditions affecting the wall.

The fixed humidity chamber consisted of a large plastic container filled a third of the way with water. Metal racks were set in the container so that the resting stone cubes would avoid coming in direct contact with the water. The container was then sealed with tape to allow the relative humidity to reach 100%. The samples were left in the humidity chamber for 3 days before being placed in the desiccator. The drying period consisted equally of 3 days. The 3-day alternating cycles were repeated for two weeks, totaling in 6 days of wetting and 6 days of drying (Appendix G).

4.4 d - Poulticing

Following the accelerated weathering conditions, all of the 36 stone cubes were poulticed using paper pulp. Some groups were brushed and poulticed with one or both types of surfactants, whereas some groups were only poulticed using one type of solution. All 3 stones in each group were always treated and poulticed in the same manner. Listed below are treatments performed on each group of stones.

Table IV.7 Treatment preparation.

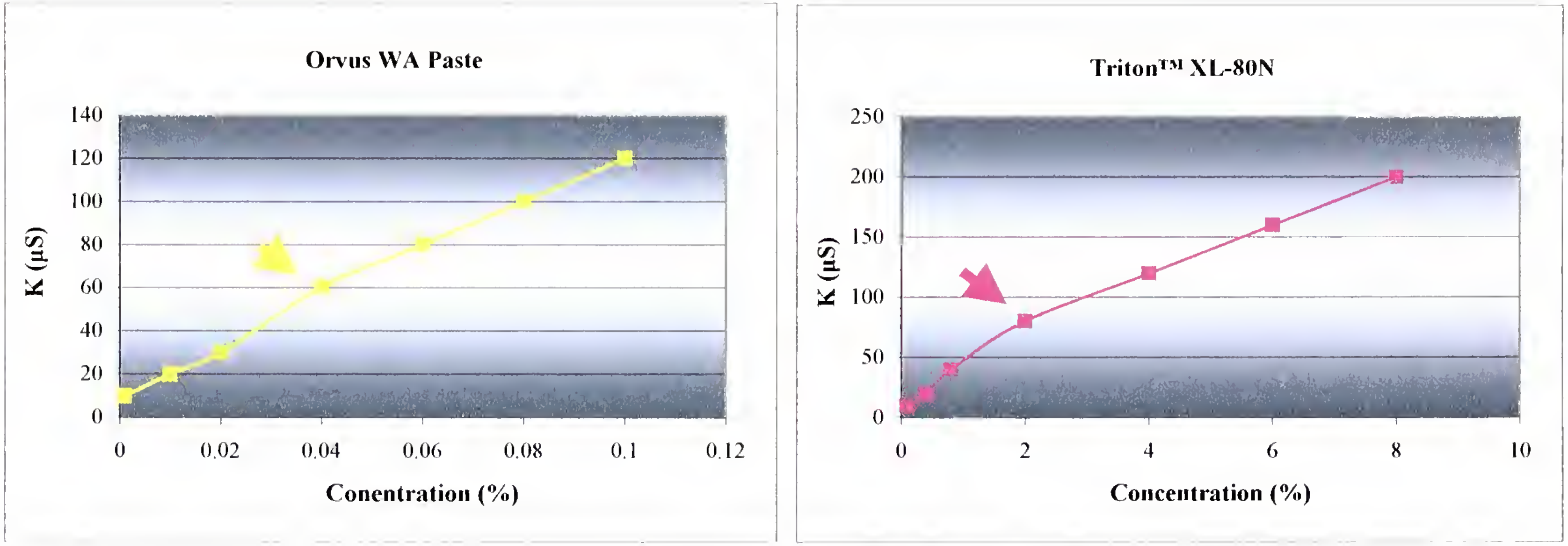
group	sample no.	salt impregnated	brushed with	poulticed with
A	1 2 3	no	none	Triton™ XL-80N
B	4 5 6	no	none	H2O
C	7 8 9	no	none	Orvus WA Paste
D	10 11 12	yes	Triton™ XL-80N	H2O
E	13 14 15	yes	Orvus WA Paste	H2O
F	16 17 18	yes	none	H2O
G	19 20 21	yes	none	Orvus WA Paste
H	22 23 24	yes	Orvus WA Paste	Orvus WA Paste
I	25 26 27	yes	Orvus WA Paste	Triton™ XL-80N
J	28 29 30	yes	none	Triton™ XL-80N
K	31 32 33	yes	Triton™ XL-80N	Triton™ XL-80N
L	34 35 36	yes	Triton™ XL-80N	Orvus WA Paste

The concentrations of surfactants used were 2% Triton™ XL-80N and 0.04% Orvus. This was based on the slope of their conductance curves. For each type of surfactant, seven different concentrations were prepared and measured for their conductance (Table IV.8). A detailed explanation of conductimetric measurements will be discussed in Section *4.4 e*. From the graph, it was possible to determine the point of critical micelle concentration, which is where the changes in the slope occur. Since the effectiveness of the surfactant does not increase above its critical micelle concentration (refer to Chapter III), the correlating concentrations (%) were chosen for both surfactants (Graph IV.4).

Table IV.8 Conductimetric measurements for Orvus and Triton.

Orvus WA Paste	concentration (%)	0.001	0.01	0.02	0.04	0.06	0.08	0.1
	conductance (µS)	10	20	30	60	80	100	120
Triton™ XL-80N	concentration (%)	0.1	0.4	0.8	2.0	4.0	6.0	8.0
	conductance (µS)	10	20	40	80	120	160	200

Graph IV.4 Slope change indicates critical micelle concnetration (CMC) point.



For the groups that were brushed with the surfactants, a total of 10 applications were performed over a 25-minute period. Each application consisted of using a 2" wide painter's brush where the aqueous solution of respective surfactants was applied once to all six sides of the sample cubes. First 5 applications were made in 1-minute intervals to reflect the rate at which the stone cubes absorbed the surfactants. By the fifth application, however, the rate of absorption had decreased and residual bubbles of surfactant remained on the surfaces of the cubes for a few minutes. Therefore, the subsequent applications were made in 5-minute intervals. Following the final application, the cubes were left to air-dry for an hour at ambient temperature and humidity.

For poulticing, each solution (Triton™ XL-80N, Orvus, and tap water) was added to the paper pulp mixture until suitable workability was achieved. The samples were first covered with Japanese tissue paper (saturated with respective solution) to keep the paper pulp from adhering to the stone's surface. The poultice was then applied to all six sides of the cubes and wrapped individually in plastic wrap to prevent evaporation of the solutions.

Fig. IV.6 A sample cube covered with Japanese tissue paper and paper pulp poultice.

After two days, each poultice was removed and placed in a beaker. They were initially dried in an oven at approximately 65 °C (Appendix H). Due to an unfortunate accident in the lab, the temperature for one of the ovens was

Fig. IV.7 Samples poulticed and wrapped in plastic wrap.

raised to 160 °C for an indeterminate amount of time. Some of the samples, therefore, experienced various degrees of burning and are indicated accordingly in the appendices. Once completely dried, the poultices were analyzed for their salt content. The amount of salt extracted were determined using: "Quantab" NaCl titrator strips manufactured by HACH® and the "pH/Conductance Pocket Pal™" meter manufactured by Omega®.

The methodology described above also applies to the second round of poulticing performed. Variations in the preparation include the omission of any solution-brushing as well as limiting the aqueous solution to deionized water in the paper pulp mixture.

4.4 e - Results

The dried poultices were each immersed in 0.4 (L) of deionized water for 1 hour and filtered. The filtered solutions were analyzed for their salt content using both the conductimetric measurements and titrator strips. Although the analytical strips indicate the amount of NaCl found (expressed as %w/v) per sample poulticed, the conductance measurements (expressed in μS) indicate only the conductance level present per sample poulticed. Therefore, it was necessary to convert the conductance to reflect the amount of NaCl present.

Conductivity, the ability of a solution to carry electricity due to the mobility of ions when subjected to an electric field, is the inverse of resistance. Conductance (*K*) of a solution, therefore, depends on the concentration of the mobile ions and the

conductimetric measurements are most suited for strong electrolytes such as sodium chloride.[86]

Since conductance (*K*) varies greatly with concentration of mobile ions and temperature, a known standard solution was used to verify the accuracy of the "Pocket Pal™" meter during the testing period under ambient lab conditions. With standard solutions, the calibration curves were obtained. That is, the amount of conductance was correlated to molarity so that the curves can be used to estimate the salt concentrations (Table IV.9). While Fisher Scientific Co. supplied the standard potassium chloride solutions, the standard sodium chloride solutions were prepared in the laboratory. It is evident from Graph IV.4 that there is a linear relationship between molar concentration and conductance.

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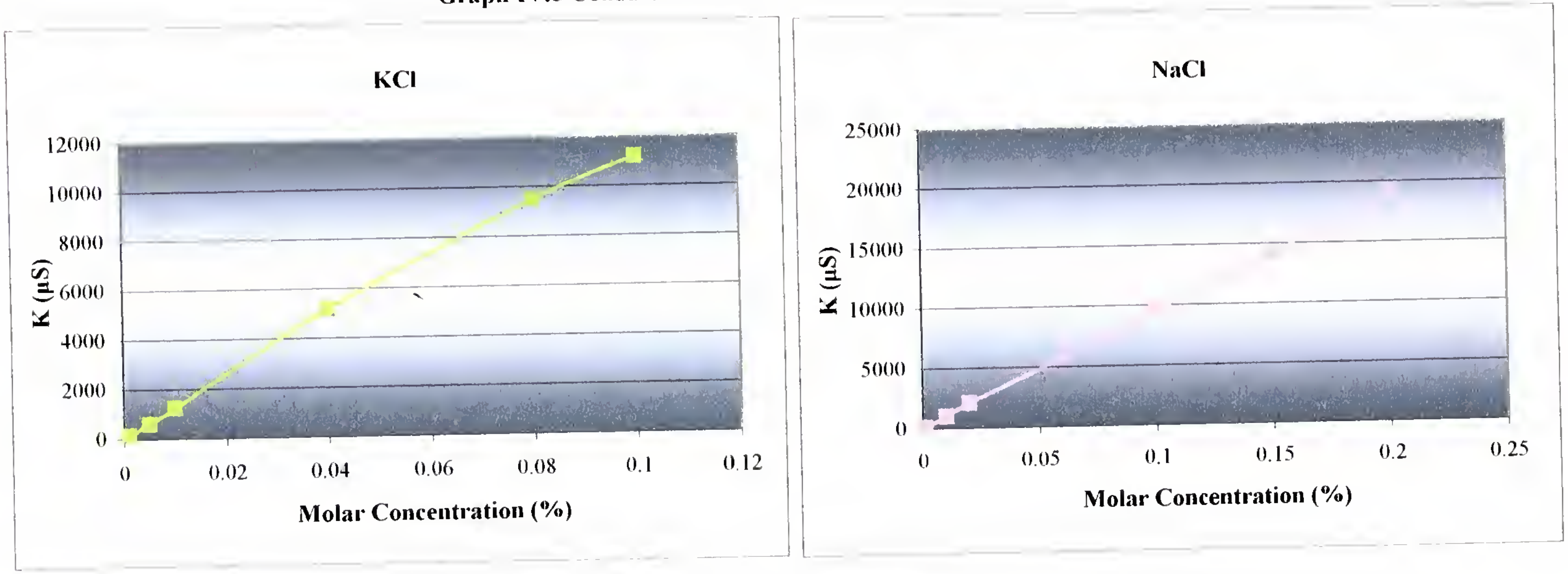
[86] J. Freedland, *Soluble Salts in Porous Materials: Evaluating Effectiveness of their Removal*, (master's thesis, University of Pennsylvania, 1999), p.36.

Table IV.9 Conductimetric measurements for KCl and NaCl.

KCl	molar concentration (%)	0.001	0.005	0.01	0.04	0.08	0.1	
	conductance (µS)	140	610	1260	5160	9470	11190	
NaCl	molar concentration (%)	0.001	0.01	0.02	0.06	0.1	0.15	0.2
	conductance (µS)	100	970	2120	5750	9570	14190	19270
If $m = (y-b)/x$ and $b = 0$, then for every molar concentration m =		100000	97000	106000	95833	95700	94600	96350
average value of m =		**97926.2**						

Graph IV.5 Conductance standardization curve for KCl and NaCl.



Examining the calibration curve of NaCl, the line was assumed to start at (0,0) and increased as molar concentration increased. Given these known molar concentrations and their corresponding conductance, the slope of the line (*m)* was calculated as follows:

$$m = (y-b) / x$$

where, $b = 0$

x = molar concentration

y = conductance

Once the *m* value for all calibration points was obtained, its average (97926.2) was taken and used to determine the unknown molar concentrations. For example, the unknown *x* can be calculated with the formula: $x = (y - b) / m$. With the calculated molarity, the amount of extracted NaCl can be derived using the salt's molecular weight and the amount of water added. Therefore, a filtered solution (#1) with a conductance measurement of 4190 (μS) can be calculated as follows:

$$\textit{Molar concentration (g/L)} = [K\,(\mu S) - 0] / m$$
$$= [4190\,(\mu S) - 0] / 97926.2$$
$$= 0.0428$$

then,

$$\textit{wt. of salt extracted (g/L)} = \textit{Molar concentration (mol/L)} \bullet \textit{M.W. of NaCl (g/mol)}$$
$$= 0.0428\,(\text{mol/L}) \bullet 58.44\,(\text{g/mol})$$
$$= 2.50\,(\text{g/L})$$

then,

$$\textit{wt. of salt extracted (g)} = \textit{wt. of salt extracted (g/L)} \bullet \textit{amount of } H_2O \textit{ added (L)}$$
$$= 2.50\,(\text{g/L}) \bullet 0.4\,(\text{L})$$
$$= 1.00\,(\text{g})$$

If the concentration measured was at or above the maximum value of the meter, it was necessary to dilute the filtered solution to a known volume. All dilutions were made with the same amount of solution - 0.05 (L) of sample solution was diluted to 0.1 (L) - and the following factor was added to the above formula:

diluted volume (L) / measured sample solution (L)

For example, Sample 10 had an initial conductance measurement greater than 20000 (µS), a value beyond the range of the "Pocket Pal™." Therefore, 0.05 (L) of the filtered solution sample was diluted to 0.1 (L), which had a measurement of 11280 (µS). Based on the second conductimetric value and the known amount of dilution, the weight of the salt extracted was obtained as such:

wt. of salt extracted (g) = [K (µS) – 0] / m (g/L) • M.W. of NaCl (g) • amount of H_2O added (L) • diluted volume (L) / measured sample solution (L)

therefore,

wt. of salt extracted (g) = [11280 (µS) - 0] / 97926.2 (g/L) • 58.44 (g) • 0.4 (L) • 0.1 (L) / 0.05 (L)
= 5.385 (g)

The same calculations were also applied to the surfactant solutions and tap water. Conductimetric measurement for Triton was 75 (µS), 50 (µS) for Orvus, and 330 (µS) for tap water. The corresponding weight of NaCl from each respective solution was calculated as follows:

wt. of NaCl from Triton (g) = [K (µS) – 0] / m (g/L) • M.W. of NaCl (g) • amount of H_2O added (L)
= [75 – 0] / 97926.2 (g/L) • 58.44 (g) • 0.4 (L)
= 0.0179 (g)

For each sample, the sum of the corresponding weight of NaCl from tap water and from the respective poultice solutions was subtracted from the obtained weight of salt extracted and expressed as (%w/w). That is, the final weight (g) of the sample was divided by the cube's initial dry weight and multiplied by 100. For example, Sample 1 weighed 287.57 (g) prior to being poulticed with Triton solution (Appendix J). Therefore,

NaCl extracted (%w/w) = wt. of salt extracted (g) - [wt. of NaCl from Triton (g) + wt. of NaCl from tap H_2O (g)] / wt. of dry sample cube (g) • 100

= 1.00 (g) – [0.0179 (g) + 0.0787 (g)] / 287.57 (g) •100

= 0.31 (%w/w)

Since the amount of NaCl extracted was expressed as (%w/v) by "Quantab®," all measurements indicated by the titrator strips were converted from (%w/v) to (w/v) for consistency. Again, if the concentration obtained for a sample was at the maximum value of the strip indicator, it was necessary to dilute the filtered solution to a known volume. All dilutions were performed in the same manner as described above. For example, the amount of NaCl extracted from Sample 10 was indicated as greater than 0.01036 (g/mL). Therefore, 50.00 (mL) of the filtered solution sample were diluted to 100.00 (mL), which then had a measurement of 0.00673 (g/mL). The amount of salt extracted (%w/w) was calculated as follows:

wt. of salt extracted (g) = wt. of salt extracted (g/mL) • amount of H_2O added (mL) • diluted volume (mL) / measured sample solution (mL)

= 0.00673 (g/mL) • 400 (mL) • 100 (mL) / 50 (mL)

= 5.384 (g)

then,

NaCl extracted (%w/w) = wt. of salt extracted (g) / wt. of dry sample cube (g) • 100

= 5.384 (g) / 280.56 (g) • 100
= 1.919 (%w/w)

In the case of the analytical strips, the weight of NaCl found in the surfactant solutions and tap water was below 0.046 (%w/w) and consequently considered negligible. As a result, there was no need to account for any amount of NaCl other than what was measured from the solutions. Once the amount of NaCl extracted (%w/w) was determined for each sample, an average of the three samples and their standard deviation was calculated to represent an average for the group (Appendix N).

Given the known concentration of NaCl originally present in the stone and the amount introduced during the experiment, it was possible to calculate the total amount of salt found per sample. Similarly, by quantifying the extracted salt for both rounds of poulticing, it was possible to calculate the total amount of salt extracted per sample. Based on these known values, it was possible to calculate the % NaCl removed per treatment type and evaluate the effectiveness of the different methodology applied. Table IV. 10 and Table IV.11 compares the results obtained from the two types of quantitative analyses.

The reason for any discrepancies between the values obtained by the two different analytical techniques is due to the poor results provided by Quantab when a value falls near the extreme ends of the titrator's supported range. Also, the corrections made for the conductimetric measurements for tap water and for surfactant are only approximations.

Nonetheless, the results of either method of measurement show the same trends, particularly for the salt-impregnated samples (Graph IV.6 and IV.7).

TABLE IV.10 NaCl extraction results based on "Pocket Pal."

The average value of NaCl inherent in stone (%w/w) was calculated in Table IV.5. The average value of NaCl introduced (%w/w) is calculated in Table IV.9.

		A	B	C	D	E	F	G
treatment type	sample no.	average NaCl originally in stone (%w/w)	average NaCl introduced (% w/w)	total NaCl in stone (%w/w) [A+B]	avg. NaCl obtained from the 1st round of poulticing (% w/w)	avg. NaCl obtained from the 2nd round of poulticing (% w/w)	total NaCl poulticed (%w/w) [D+E]	% NaCl recmoved [(F/C)*100]
Untreated Samples Poulticed with H2O	4 5 6	2.47	---	2.47	0.232	0.357	0.589	23.85
Untreated Samples Poulticed with Orvus	7 8 9	2.47	---	2.47	0.112	0.449	0.561	22.71
Untreated Samples Poulticed with Triton	1 2 3	2.47	---	2.47	0.317	0.306	0.623	25.22
Salt-impregnated Samples Poulticed with H2O	16 17 18	2.47	1.720	4.19	1.666	0.721	2.387	56.97
Salt-impregnated Samples Poulticed with Orvus	19 20 21	2.47	1.568	4.038	1.719	1.037	2.756	68.25
Salt-impregnated Samples Poulticed with Triton	28 29 30	2.47	1.358	3.828	1.752	1.045	2.797	73.07
Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13 14 15	2.47	1.419	3.889	1.108	0.725	1.833	47.13
Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22 23 24	2.47	1.503	3.973	1.406	0.844	2.250	56.63
Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25 26 27	2.47	1.553	4.023	1.484	0.866	2.350	58.41
Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10 11 12	2.47	1.525	3.995	1.820	0.854	2.674	66.93
Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34 35 36	2.47	1.650	4.12	1.310	0.849	2.159	52.40
Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31 32 33	2.47	1.648	4.118	1.372	0.775	2.147	52.14

TABLE IV.11 NaCl extraction results based on "Quantab."

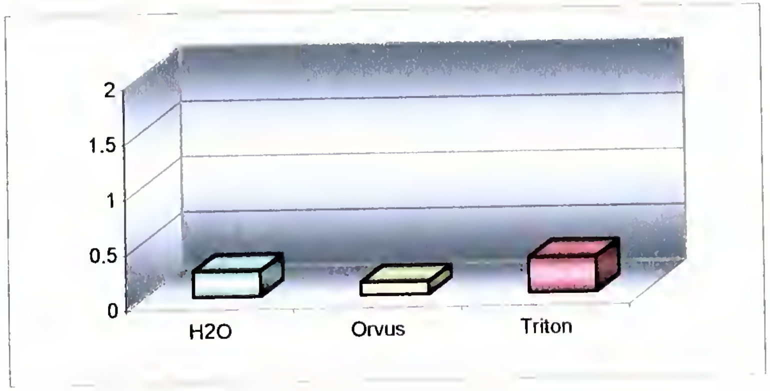
The average value of NaCl inherent in stone (% w/w) was calculated in Table IV.3. The average value of NaCl introduced (% w/w) was calculated in Table IV.6

		A	B	C	D	E	F	G
treatment type	sample no.	average NaCl originally in stone (%w/w)	average NaCl introduced (% w/w)	total NaCl in stone (%w/w) [A+B]	avg. NaCl obtained from the 1st round of poulticing (% w/w)	avg. NaCl obtained from the 2nd round of poulticing (% w/w)	total NaCl poulticed (%w/w) [D+E]	% NaCl recmoved [(F/C)*100]
Untreated Samples Poulticed with H2O	4 5 6	2.47	---	2.47	0.124	0.092	0.216	8.74
Untreated Samples Poulticed with Orvus	7 8 9	2.47	---	2.47	0.078	0.276	0.354	14.33
Untreated Samples Poulticed with Triton	1 2 3	2.47	---	2.47	0.000	0.000	0.000	0.00
Salt-impregnated Samples Poulticed with H2O	16 17 18	2.47	1.720	4.19	1.604	0.437	2.041	48.71
Salt-impregnated Samples Poulticed with Orvus	19 20 21	2.47	1.568	4.038	1.553	0.945	2.498	61.86
Salt-impregnated Samples Poulticed with Triton	28 29 30	2.47	1.358	3.828	1.492	1.115	2.607	68.10
Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13 14 15	2.47	1.419	3.889	0.923	0.482	1.405	36.13
Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22 23 24	2.47	1.503	3.973	1.274	0.651	1.925	48.45
Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25 26 27	2.47	1.553	4.023	1.253	0.691	1.944	48.32
Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10 11 12	2.47	1.525	3.995	1.722	0.739	2.461	61.60
Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34 35 36	2.47	1.650	4.12	1.034	0.595	1.629	39.54
Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31 32 33	2.47	1.648	4.118	1.150	0.523	1.673	40.63

Graph IV.6 Average amount of NaCl extracted (%w/w) per treatment type - measured with "Pocket Pal."

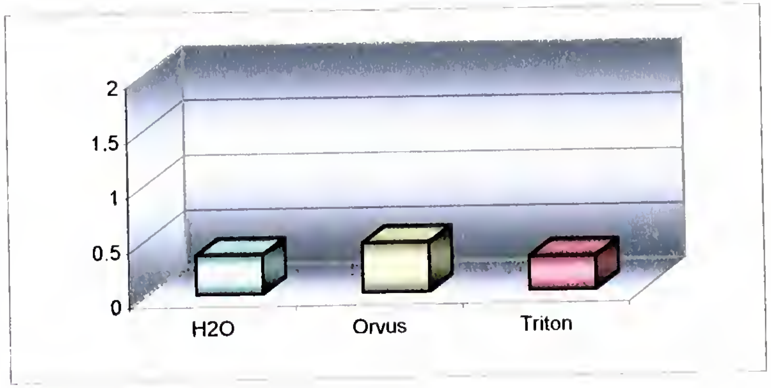
Poultice - ROUND 1 for A, B, and C.

IV.6 a Untreated samples poulticed with respective solutions



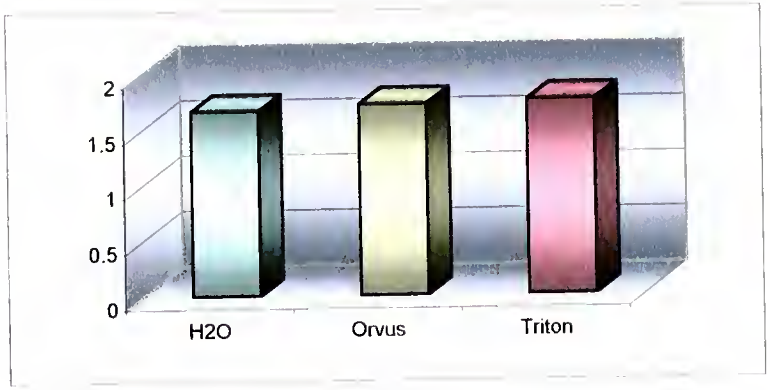
Poultice - ROUND 2 for A, B, and C.

IV.6 b Untreated samples poulticed with respective solutions



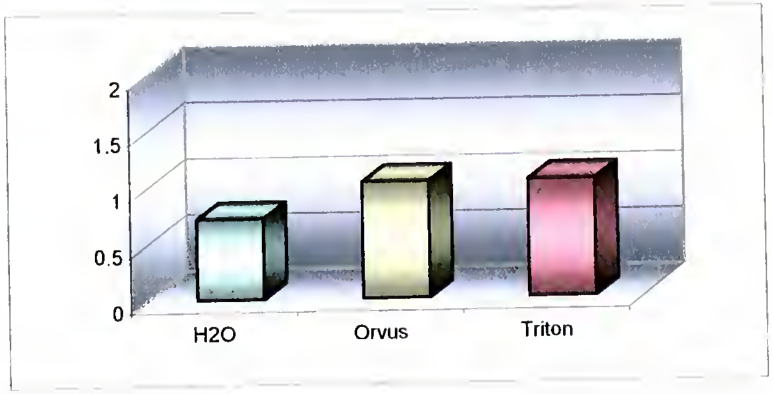
Poultice - ROUND 1 for F, G, and J.

IV.6 c Salt-impregnated samples poulticed with respective solutions



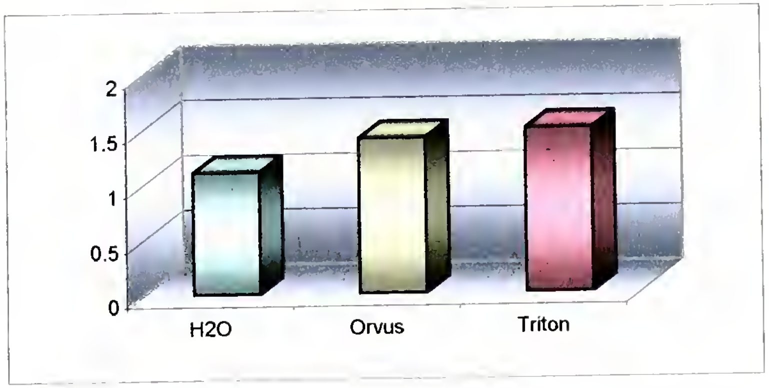
Poultice - ROUND 2 for F, G, and J.

IV.6 d Salt-impregnated samples poulticed with respective solutions



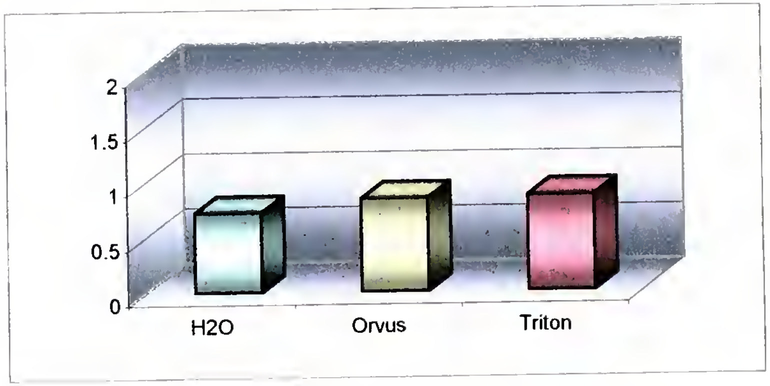
Poultice - ROUND 1 for E, H, and I.

IV.6 e Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



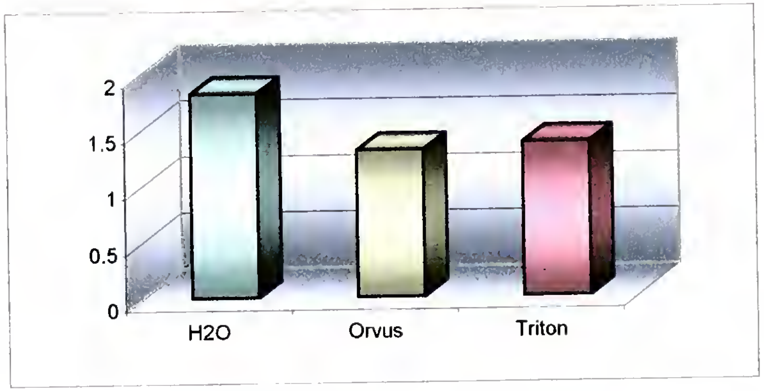
Poultice - ROUND 2 for E, H, and I.

IV.6 f Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



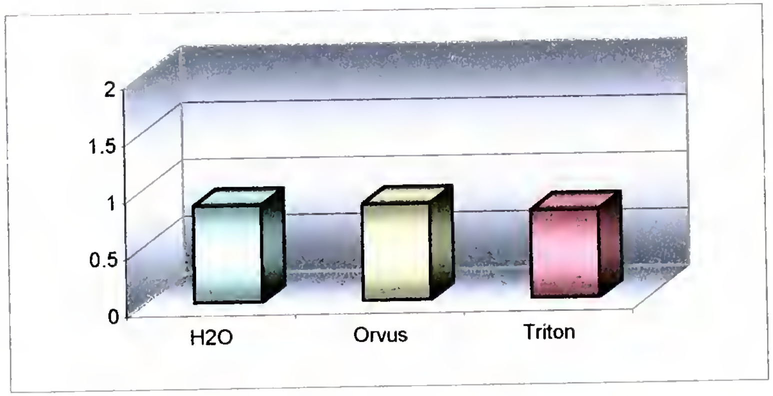
Poultice - ROUND 1 for D, L, and K.

IV.6 g Salt-impregnated samples brushed with Triton and poulticed with respective solutions



Poultice - ROUND 2 for D, L, and K.

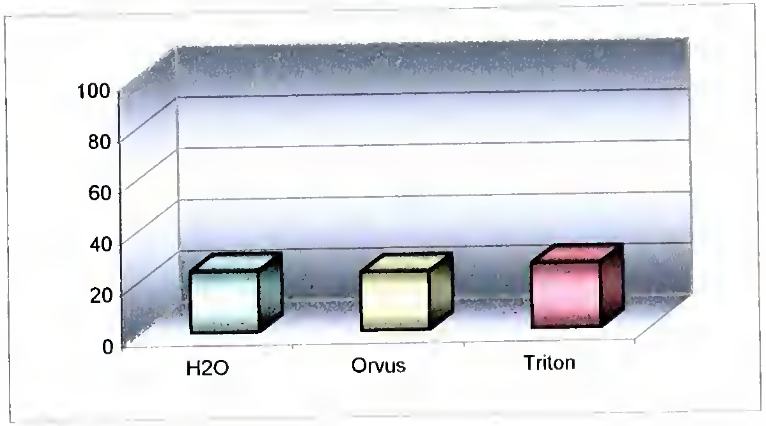
IV 6 h Salt-impregnated samples brushed with Triton and poulticed with respective solutions



% NaCl removed per treatment type

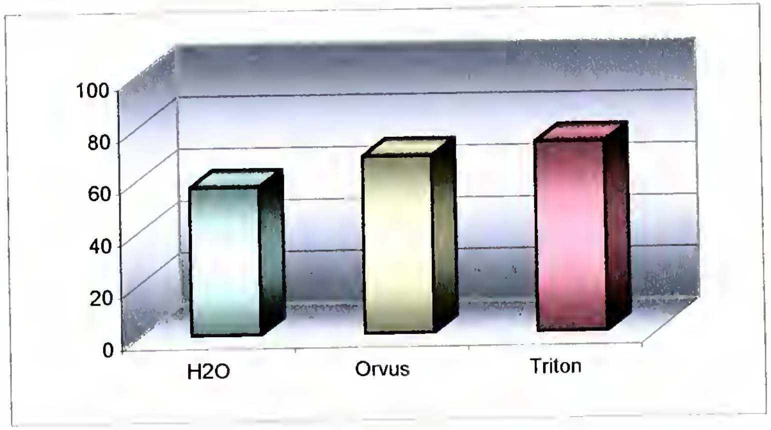
A, B, and C.

IV 6 i Untreated samples poulticed with respective solutions



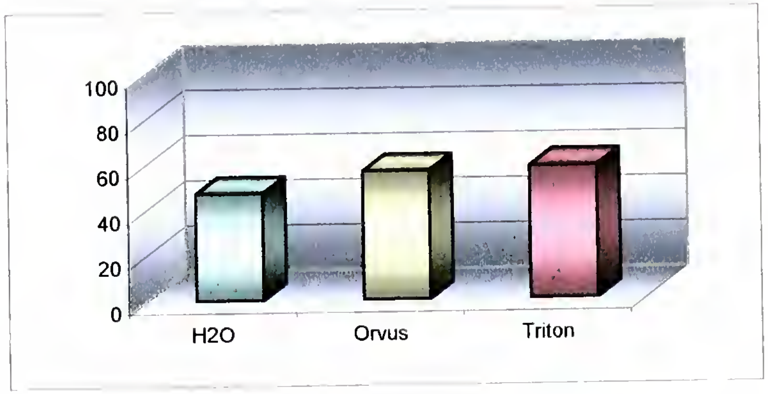
F, G, and J.

IV 6 j Salt-impregnated samples poulticed with respective solutions



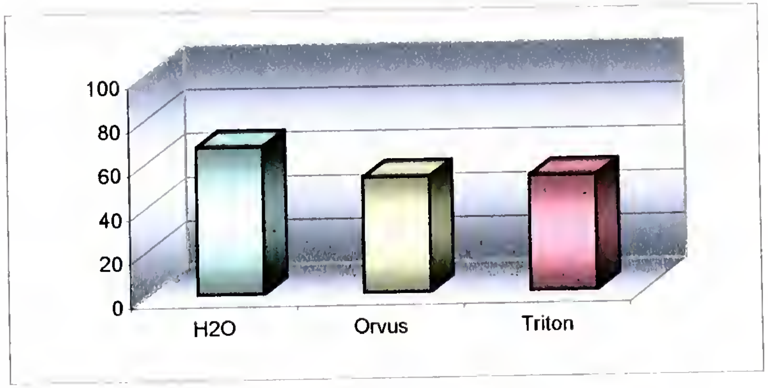
E, H, and I.

IV.6 k Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



D, L, and K.

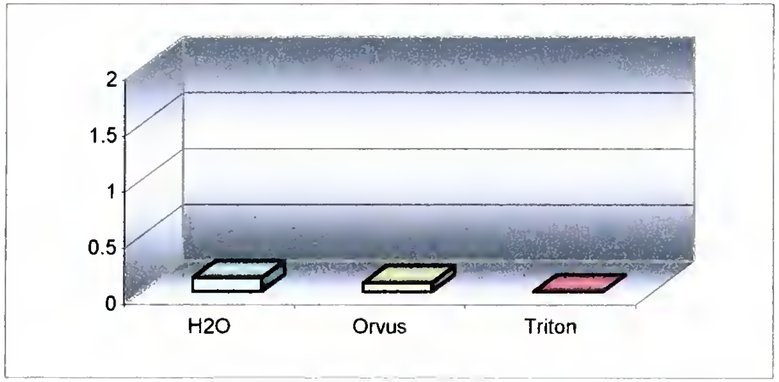
IV.6 l Salt-impregnated samples brushed with Triton and poulticed with respective solutions



Graph IV.7 Average amount of NaCl extracted (%w/w) per treatment type - measured with "Quantab."

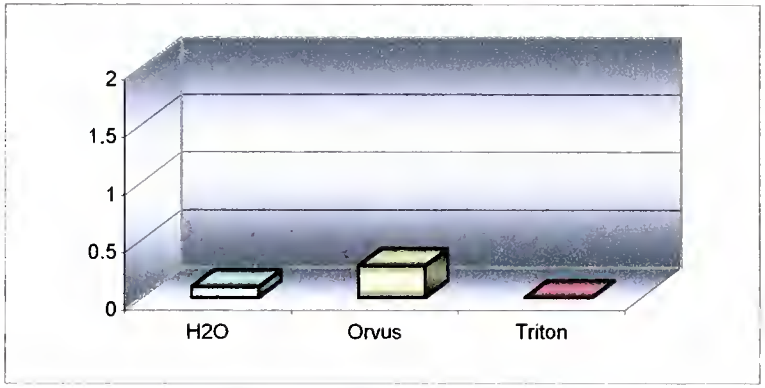
Poultice - ROUND 1 for A, B, and C.

IV.7 a Untreated samples poulticed with respective solutions



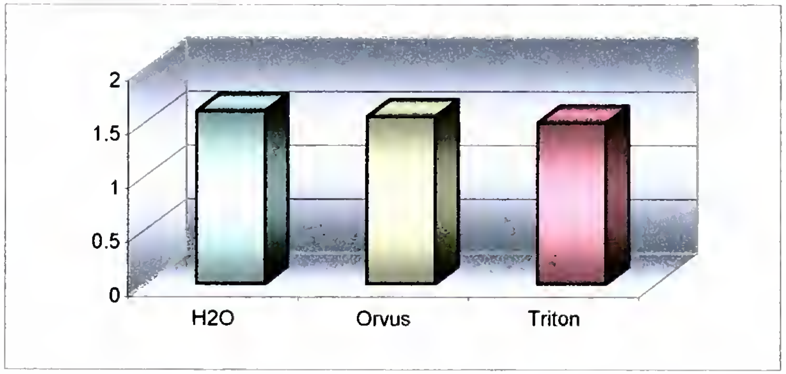
Poultice - ROUND 2 for A, B, and C.

IV.7 b Untreated samples poulticed with respective solutions



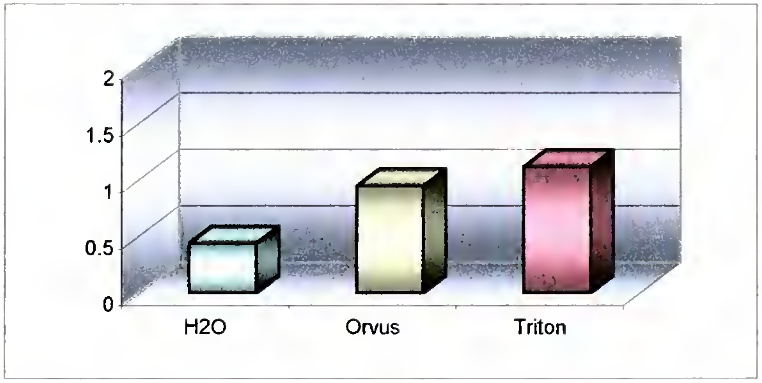
Poultice - ROUND 1 for F, G, and J.

IV.7 c Salt-impregnated samples poulticed with respective solutions



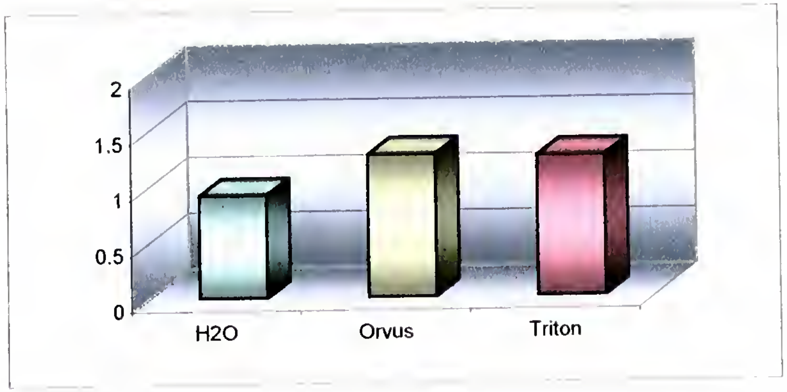
Poultice - ROUND 2 for F, G, and J.

IV 7 d Salt-impregnated samples poulticed with respective solutions



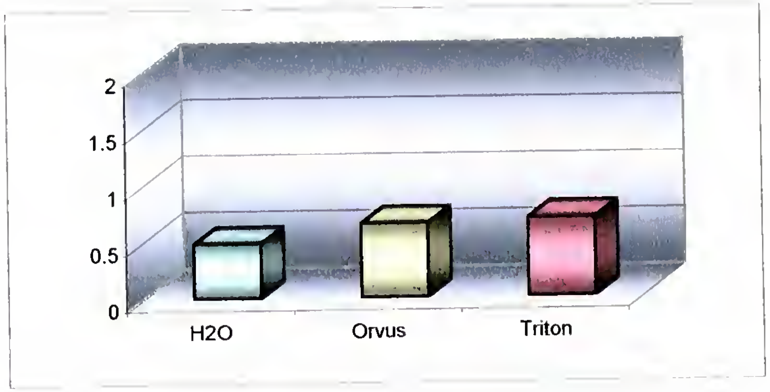
Poultice - ROUND 1 for E, H, and I.

IV.7 e Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



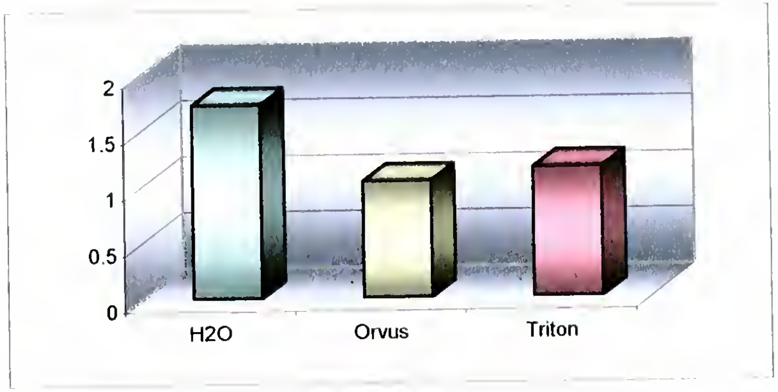
Poultice - ROUND 2 for E, H, and I.

IV.7 f Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



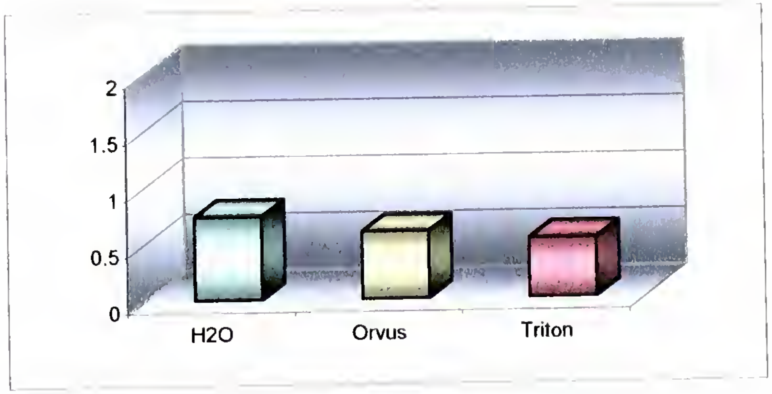
Poultice - ROUND 1 for D, L, and K.

IV.7 g Salt-impregnated samples brushed with Triton and poulticed with respective solutions



Poultice - ROUND 2 for D, L, and K.

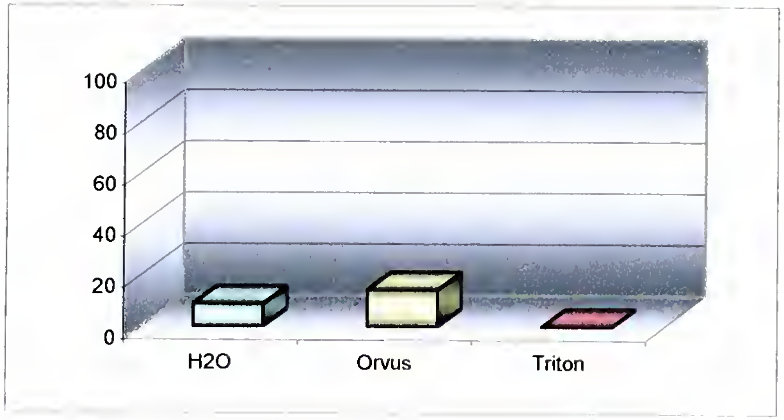
IV.7 h Salt-impregnated samples brushed with Triton and poulticed with respective solutions



% NaCl removed per treatment type

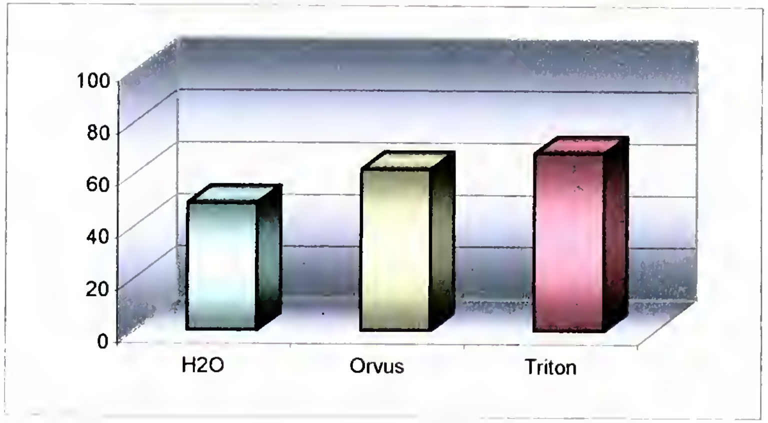
A, B, and C.

IV.7 i Untreated samples poulticed with respective solutions



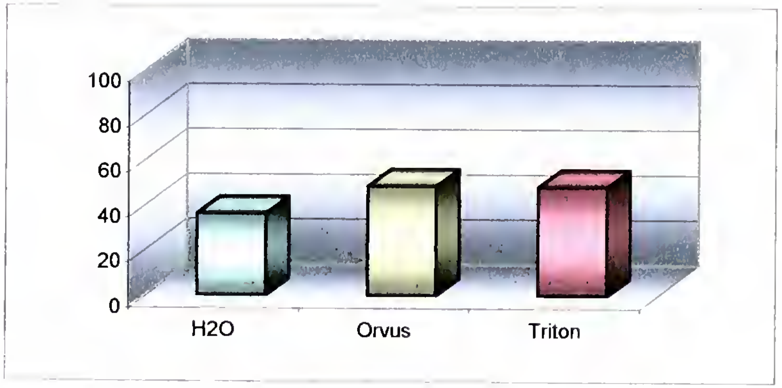
F, G, and J.

IV.7 j Salt-impregnated samples poulticed with respective solutions



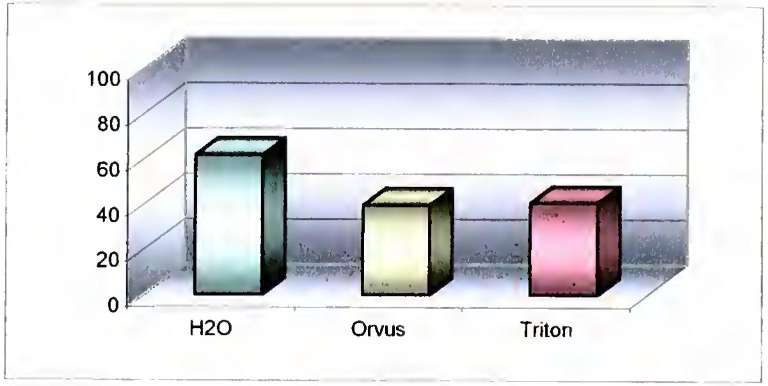
E, H, and I.

IV.7 k Salt-impregnated samples brushed with Orvus and poulticed with respective solutions



D, L, and K.

IV.7 l Salt-impregnated samples brushed with Triton and poulticed with respective solutions



CHAPTER V – CONCLUSION

Based on the stone characterization, it has been found that the currently proposed replacement stone has relatively high clay (10%) and soluble-salt content (5%). Therefore, the use of surfactant at first appears to be a reasonable option for controlling clay swelling and aiding in desalination. However, when tested, the results indicate that the presence of the surfactant does not significantly enhance salt-extraction.

Data from the 1st round of poulticing show more dispersed results compared to the results obtained from the 2nd round of poulticing. This is probably due to the fact that the 1st round of poulticing mostly removes the accumulated salts on the surface. However, such action pulls the salts from deeper within the stone, resulting in a better salt distribution for the 2nd round of poulticing.

In general, Triton™ XL-80N appears to produce more consistent results than the Orvus WA paste as reflected by their standard deviations. For the brushed samples, it has been found that pre-brushing with Orvus does not improve extraction when poulticed with water, unlike pre-brushing with Triton and poulticing with water. Also, samples brushed with Orvus and poulticed with Triton show more consistent results than when the order is reversed.

Data from the 2nd round of poulticing for the untreated stones show that using Orvus in the poultice is more efficient for removing salts. For the salt-impregnated samples, however, the amount of NaCl extracted by Triton is greater than the amount obtained from Orvus.

For both treated and untreated stones, there seems to be a general improvement in extraction when poulticed with either surfactant, albeit the difference observed is not very significant. In any event, the presence of surfactant in the poultice mixture enhances desalination for both rounds of poulticing, in particular for the 2nd round.

The overall salt extraction achieved by the two poultices corresponds to about 60-70% NaCl extraction for either pre-brushing with Triton and poulticing with water or poulticing with either Orvus or Triton. Brushing with a surfactant and poulticing with either the same or another surfactant does not necessarily lead to increased extraction and in some cases may actually show a decrease in extraction.

Based on the results obtained, the use of surfactant is not recommended as a preferred treatment method for the historic Ayyubid wall. Although the presence of surfactant enhances salt-extraction, the difference observed is not enough to warrant the cost incurred if chosen as a conservation treatment. However, if chosen, a more cost effective method may be to combine brushing with Triton and poulticing with water.

Also, it is important to point out that results appear to depend heavily on the types of limestone and surfactant tested. Therefore, further research is necessary if the surfactant is to be used successfully on the Ayyubid wall. Additional limestone samples should be tested using various types of surfactants in order to accurately assess the degree of improvement offered by the surfactant. In the mean time, the author concludes that given the size of the Ayyubid Wall, perhaps a better alternative to consider is a long-term desalination technique such as sacrificial renders.

APPENDICES

Appendix A - Drying data for samples a & b, following chemical analysis for salt content.

[illegible] of the crushed [illegible] added to the [illegible] and filtered to determine soluble salt content

*Soluble salt (%w/w) (wt. of original sample wt. of salt-free sample) * 100 wt. of original sample*

sample	wt. of dish (g)	wt. of filter paper (g)	dish + filter paper (g)	hours dried at 100°C						wt. of salt-free solid (g)	original wt.-salt-free wt. (g)	soluble salt (%w/w)	average (%w/w)	stdv. (±)
				24	48	72	96	120	144					
[illegible]	41.03	2.3	43.33	91.30	91.23	90.98	90.97	90.96	90.96	47.63	2.37	4.74	4.68	0.06
[illegible]	38.8	2.27	41.07	89.13	89.07	88.79	88.78	88.76	88.76	47.69	2.31	4.62		

Appendix B - Drying data for samples a & b, following HCl ingestion.

[illegible] HCl added [illegible]

*% fines (acid-insoluble residue) = (wt. of dry fines wt. of original sample) * 100*

sample	wt. of dish (g)	wt. of filter paper (g)	dish + filter paper (g)	hours dried at 100°C							wt. of dried fines (g)	% fines	average (%w/w)	stdv. (±)
				24	48	72	96	120	144	168				
a	8.48	3.72	12.20	18.53	17.62	17.42	17.39	17.30	17.29	17.30	5.10	10.20	9.28	0.92
b	8.44	3.98	12.42	17.50	17.00	16.94	16.77	16.70	16.60	16.60	4.18	8.36		

Appendix C - Initial weight of 36 Egyptian limestone cubes.

All samples dried at 60 C Weights are presented in grams

sample no.	hours				
	0	48	72	96	120
4	304.24	303.93	303.89	303.88	303.87
5	271.04	270.81	270.78	270.77	270.77
6	287.22	286.81	286.76	286.74	286.73
7	303.51	303.12	303.07	303.06	303.04
8	289.20	288.83	288.78	288.76	288.75
9	286.34	285.93	285.88	285.87	285.85
1	288.16	287.78	287.67	287.60	287.57
2	287.85	287.45	287.39	287.27	287.25
3	280.37	280.04	279.93	279.88	279.82
16	300.02	299.72	299.69	299.66	299.66
17	282.78	282.55	282.53	282.53	282.52
18	285.85	285.62	285.60	285.57	285.58
19	301.87	301.78	301.55	301.48	301.33
20	284.37	283.95	283.94	283.93	283.90
21	296.20	295.73	295.73	295.69	295.68
28	292.11	291.70	291.64	291.63	291.63
29	285.96	285.50	285.48	285.45	285.46
30	297.37	296.87	296.87	296.86	296.84
13	280.04	279.68	279.63	279.63	279.61
14	289.86	289.47	289.41	289.40	289.38
15	287.72	287.29	287.26	287.24	287.22
22	318.76	318.31	318.30	318.25	318.26
23	294.58	294.14	294.14	294.11	294.10
24	294.03	293.62	293.57	293.58	293.57
25	287.16	286.77	286.71	286.71	286.72
26	278.50	278.06	278.01	278.01	278.00
27	287.83	287.45	287.37	287.38	287.38
10	281.03	280.67	280.58	280.57	280.56
11	297.45	297.08	296.99	296.96	296.95
12	293.60	292.95	293.07	293.01	292.98
34	292.93	292.50	292.44	292.41	292.39
35	286.75	286.50	286.49	286.46	286.47
36	278.47	278.20	278.15	278.14	278.12
31	288.45	288.03	287.98	287.98	287.98
32	280.19	279.94	279.91	279.93	279.93
33	296.00	295.59	295.52	295.50	295.49

Appendix D - Capillary water absorption and pertinent calculation for samples 1,2, and 3.

mt = weight of the sample at time (g), mo = weight of the dry sample (g). Mt = mt (g) - mo (g) (S) sample in contact with the porous support (cm2) given with a 5% precision. Ut = mt-mo

sample #1

time (min)	0	1	3	5	10	15	20	30	60	90	120	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
Time (sec)	0	60	180	300	600	900	1,200	1,800	3,600	5,400	7,200	86,400	172,800	259,200	345,600	432,000	518,400	604,800	691,200
square root of time (sec)	0	7.75	13.42	17.32	24.49	30.00	34.64	42.43	60.00	73.48	84.85	293.94	415.69	509.12	587.88	657.27	720.00	777.69	831.38
(S) surface (cm2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(mo) wt. of dry sample	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57	287.57
(mt) wt. of sample #1 (g)	287.57	289.39	291.17	292.01	293.26	294.18	295.02	296.5	299.71	302.21	304.01	311.31	311.41	311.56	311.74	311.89	311.98	312.02	312.04
(Ut) amount of water absorbed	0.00	1.82	3.6	4.44	5.69	6.61	7.45	8.93	12.14	14.64	16.44	23.74	23.84	23.99	24.17	24.32	24.41	24.45	24.47
t) amount of water absorbed per unit surface (g/cm2)	0.00	0.07	0.14	0.18	0.23	0.26	0.30	0.36	0.49	0.59	0.66	0.95	0.95	0.96	0.97	0.97	0.98	0.98	0.98

sample #2

time (min)	0	1	3	5	10	15	20	30	60	90	120	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
Time (sec)	0	60	180	300	600	900	1,200	1,800	3,600	5,400	7,200	86,400	172,800	259,200	345,600	432,000	518,400	604,800	691,200
square root of time (sec)	0.00	7.75	13.42	17.32	24.49	30.00	34.64	42.43	60.00	73.48	84.85	293.94	415.69	509.12	587.88	657.27	720.00	777.69	831.38
(S) surface (cm2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(mo) wt. of dry sample	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25	287.25
(mt) wt. of sample #1 (g)	287.25	289.88	292.13	293.22	294.85	296.06	297.16	299.1	303.48	307.09	309.8	312.84	313.15	313.34	313.61	313.81	313.95	314.08	314.1
(Ut) amount of water absorbed	0.00	2.63	4.88	5.97	7.60	8.81	9.91	11.85	16.23	19.84	22.55	25.59	25.90	26.09	26.36	26.56	26.70	26.83	26.85
Mt) amount of water absorbed per unit surface (g/cm2)	0.00	0.11	0.20	0.24	0.30	0.35	0.40	0.47	0.65	0.79	0.90	1.02	1.04	1.04	1.05	1.06	1.07	1.07	1.07

sample #3

time (min)	0	1	3	5	10	15	20	30	60	90	120	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
Time (sec)	0	60	180	300	600	900	1,200	1,800	3,600	5,400	7,200	86,400	172,800	259,200	345,600	432,000	518,400	604,800	691,200
square root of time (sec)	0.00	7.75	13.42	17.32	24.49	30.00	34.64	42.43	60.00	73.48	84.85	293.94	415.69	509.12	587.88	657.27	720.00	777.69	831.38
(S) surface (cm2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(mo) wt. of dry sample	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82	279.82
(mt) wt. of sample #1 (g)	279.82	281.62	282.93	283.54	284.52	285.24	285.91	287.13	289.82	292.02	293.65	301.44	301.63	301.76	301.91	302.00	302.06	302.09	302.11
(Ut) amount of water absorbed	0.00	1.80	3.11	3.72	4.70	5.42	6.09	7.31	10.00	12.20	13.83	21.62	21.81	21.94	22.09	22.18	22.24	22.27	22.29
Mt) amount of water absorbed per unit surface (g/cm2)	0.00	0.07	0.12	0.15	0.19	0.22	0.24	0.29	0.40	0.49	0.55	0.86	0.87	0.88	0.88	0.89	0.89	0.89	0.89

Appendix E - Drying results and calculations following total immersion for samples 1, 2, and 3.

= (Mmax) wt of the sample imbibed with water at the end of the test (g) - (Maf) wt of the dried sample after test * (100), Uo = total water content, Y = Ut / Uo, Q = Ut / Wd, Ψ = Ut / Volume of sample (g/cm3), Ut = mi (g.) - mo (g.), ΔT = difference in time bet. two measurements, ΔY = difference in relative moisture content, ΔM/M (%) or % porosity = Ut / mo *(100)

	sample #1																			
time (hrs)	0	0.17	0.33	0.5	1	1.5	2	3	4	5	6	8	24	48	72	96	120	144	168	192
time (min)	0	10	20	30	60	90	120	180	240	300	360	480	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
ΔT (hrs)	0	0.17	0.16	0.17	0.50	0.50	0.50	1.00	1.00	1.00	1.00	2.00	16.00	24.00	24.00	24.00	24.00	24.00	24.00	24.00
surface (cm^2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(IC) Imbibition capacity (%)	8.89	8.77	8.66	8.66	8.38	8.02	7.77	7.25	6.68	6.02	5.55	4.56	1.49	0.65	0.34	0.24	0.20	0.19	0.16	0.16
(mo) wt. of dry sample	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77	285.77
ni) wt of sample #1 at time (g)	311.17	310.83	310.53	310.53	309.73	308.68	307.97	306.5	304.86	302.98	301.62	298.79	290.02	287.64	286.73	286.46	286.33	286.31	286.23	286.22
(Uo) total water content (g)	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4	25.4
(Ut) water content (g)	25.4	25.06	24.76	24.76	23.96	22.91	22.2	20.73	19.09	17.21	15.85	13.02	4.25	1.87	0.96	0.69	0.56	0.54	0.46	0.45
(Q%) residual water content	100.00	98.66	97.48	97.48	94.33	90.20	87.40	81.61	75.16	67.76	62.40	51.26	16.73	7.36	3.78	2.72	2.20	2.13	1.81	1.77
M%) the amount of absorbed water of the sample at time	8.89	8.77	8.66	8.66	8.38	8.02	7.77	7.25	6.68	6.02	5.55	4.56	1.49	0.65	0.34	0.24	0.20	0.19	0.16	0.16
amount of water absorbed per unit area (g/cm2)	1.02	1.00	0.99	0.99	0.96	0.92	0.89	0.83	0.76	0.69	0.63	0.52	0.17	0.07	0.04	0.03	0.02	0.02	0.02	0.02
(Y) relative moisture content	1.00	0.99	0.97	0.97	0.94	0.90	0.87	0.82	0.75	0.68	0.62	0.51	0.17	0.07	0.04	0.03	0.02	0.02	0.02	0.02
ΔY	0.00	0.01	0.01	0.00	0.03	0.04	0.03	0.06	0.06	0.07	0.05	0.11	0.35	0.09	0.04	0.01	0.01	0.00	0.00	0.00
ΔT) amount of moisture lost per unit time	---	0.08	0.07	0.07	0.06	0.08	0.06	0.06	0.06	0.07	0.05	0.06	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00
(Ψ) moisture content (g/cm3)	0.203	0.200	0.198	0.198	0.192	0.183	0.178	0.166	0.153	0.138	0.127	0.104	0.034	0.015	0.008	0.006	0.004	0.004	0.004	0.004

	sample #2																			
time (hrs.)	0	0.17	0.33	0.5	1	1.5	2	3	4	5	6	8	24	48	72	96	120	144	168	192
time (min)	0	10	20	30	60	90	120	180	240	300	360	480	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
ΔT (hrs)	0	0.17	0.16	0.17	0.50	0.50	0.50	1.00	1.00	1.00	1.00	2.00	16.00	24.00	24.00	24.00	24.00	24.00	24.00	24.00
surface (cm^2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
(IC) Imbibition capacity (%)	9.53	9.41	9.30	9.16	9.02	8.64	8.40	7.88	7.30	6.63	6.12	5.03	1.37	0.47	0.21	0.18	0.15	0.16	0.14	0.14
(mo) wt of dry sample	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67	286.67
(mi) wt of sample #1 at time (g)	314.00	313.64	313.33	312.92	312.52	311.44	310.75	309.26	307.59	305.67	304.22	301.08	290.60	288.03	287.26	287.18	287.11	287.13	287.07	287.06
(Uo) total water content (g)	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.35	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.33	27.33
(Ut) water content (g)	27.33	26.97	26.66	26.25	25.85	24.77	24.08	22.59	20.92	19.00	17.55	14.41	3.93	1.36	0.59	0.51	0.44	0.46	0.40	0.39
(Q%) residual water content	100.00	98.68	97.55	96.05	94.58	90.63	88.11	82.66	76.55	69.52	64.22	52.73	14.38	4.98	2.16	1.87	1.61	1.68	1.46	1.43
ΔM%) the amount of absorbed water of the sample at time	9.53	9.41	9.30	9.16	9.02	8.64	8.40	7.88	7.30	6.63	6.12	5.03	1.37	0.47	0.21	0.18	0.15	0.16	0.14	0.14
A) amount of water absorbed per unit area (g/cm2)	1.09	1.08	1.07	1.05	1.03	0.99	0.96	0.90	0.84	0.76	0.70	0.58	0.16	0.05	0.02	0.02	0.02	0.02	0.02	0.02
(Y) relative moisture content	1.00	0.99	0.98	0.96	0.95	0.91	0.88	0.83	0.77	0.70	0.64	0.53	0.14	0.05	0.02	0.02	0.02	0.02	0.01	0.01
ΔY	0.00	0.01	0.01	0.02	0.01	0.04	0.03	0.05	0.06	0.07	0.05	0.11	0.38	0.09	0.03	0.00	0.00	0.00	0.00	0.00
/ΔT) amount of moisture lost per unit time	---	0.08	0.07	0.09	0.03	0.08	0.05	0.05	0.06	0.07	0.05	0.06	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00
(Ψ) moisture content (g/cm3)	0.219	0.216	0.213	0.210	0.207	0.198	0.193	0.181	0.167	0.152	0.140	0.115	0.031	0.011	0.005	0.004	0.004	0.004	0.003	0.003

	sample #3																			
time (hrs)	0	0.17	0.33	0.5	1	1.5	2	3	4	5	6	8	24	48	72	96	120	144	168	192
ime (min)	0	10	20	30	60	90	120	180	240	300	360	480	1,440	2,880	4,320	5,760	7,200	8,640	10,080	11,520
ΔT (hrs)	0	0.17	0.16	0.17	0.50	0.50	0.50	1.00	1.00	1.00	1.00	2.00	16.00	24.00	24.00	24.00	24.00	24.00	24.00	24.00
urface (cm^2)	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
ibition capacity (%)	8.47	8.35	8.25	8.11	7.97	7.60	7.36	6.85	6.28	5.62	5.14	4.15	1.42	0.61	0.31	0.22	0.17	0.17	0.14	0.13
t. of dry sample	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72	277.72
sample #1 at time (g)	301.25	300.92	300.63	300.24	299.85	298.83	298.17	296.75	295.15	293.32	292.00	289.25	281.66	279.42	278.57	278.32	278.20	278.18	278.10	278.09
l water content (g)	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53	23.53
ater content (g)	23.53	23.2	22.91	22.52	22.13	21.11	20.45	19.03	17.43	15.6	14.28	11.53	3.94	1.7	0.85	0.6	0.48	0.46	0.38	0.37
dual water content	100.00	98.60	97.37	95.71	94.05	89.72	86.91	80.88	74.08	66.30	60.69	49.00	16.74	7.22	3.61	2.55	2.04	1.95	1.61	1.57
amount of absorbed water : sample at time	8.47	8.35	8.25	8.11		7.60	7.36	6.85	6.28	5.62	5.14	4.15	1.42	0.61	0.31	0.22	0.17	0.17	0.14	0.13
of water absorbed per unit rea (g/cm2)	0.94	0.93	0.92	0.90	0.89	0.84	0.82	0.76	0.70	0.62	0.57	0.46	0.16	0.07	0.03	0.02	0.02	0.02	0.02	0.01
ve moisture content	1.00	0.99	0.97	0.96	0.94	0.90	0.87	0.81	0.74	0.66	0.61	0.49	0.17	0.07	0.04	0.03	0.02	0.02	0.02	0.02
ΔY	0.00	0.01	0.01	0.02	0.02	0.04	0.03	0.06	0.07	0.08	0.06	0.12	0.32	0.10	0.04	0.01	0.01	0.00	0.00	0.00
nt of moisture lost per unit time	---	0.08	0.08	0.10	0.03	0.09	0.06	0.06	0.07	0.08	0.06	0.06	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00
ture content (g/cm3)	0.188	0.186	0.183	0.180	0.177	0.169	0.164	0.152	0.139	0.125	0.114	0.092	0.032	0.014	0.007	0.005	0.004	0.004	0.003	0.003

Note: 0 hr. represents the time immediately following the 24-hours total immersion

sample no	asymptotical value obtained from the drying rate (g)	hours dried in an oven at 60°C			
		24	48	72	96
1	286.22	286.09	286.02	286.00	285.99
2	287.06	286.87	286.79	286.74	286.74
3	278.09	278.07	278.07		

The samples were dried in an oven to ensure complete moisture loss before treatment

Appendix F - Drying rate following 24-hr. NaCl immersion.

group	sample no.	wt. of dried sample (g)	wt. following 10 hrs. of air-drying (g)	hours dried at 105°C																
				24	48	72	96	120	144	168	192	216	240	264	288	312	336	360	384	408
F	16	299.66	324.47	319.82	318.74	318.42	318.09	317.62	317.04	316.21	315.41	314.19	313.77	312.54	311.47	304.16	304.04	304.00	304.00	304.00
	17	282.52	305.33	301.73	300.66	300.25	299.91	297.05	298.30	297.50	296.70	295.26	294.85	293.84	293.21	287.64	287.41	287.38	287.36	287.38
	18	285.58	311.42	307.77	305.97	305.64	305.29	304.07	303.34	301.98	300.91	299.39	298.89	297.66	296.90	291.40	291.30	291.28	291.27	291.27
G	19	301.33	319.59	315.67	315.27	314.92	314.53	313.59	312.09	310.63	309.54	308.27	307.81	306.62	305.94	301.28	300.67	300.56	300.55	300.54
	20	283.90	303.03	299.10	298.33	296.08	294.60	290.36	288.68	288.58	288.57	288.65	288.52	288.54	---	---	---	---	---	288.54
	21	295.68	318.51	315.18	314.38	314.00	313.63	312.96	312.30	311.51	310.69	309.50	309.10	307.88	307.03	300.57	300.12	300.06	300.04	300.06
J	28	291.63	311.60	307.37	306.35	305.88	305.45	303.56	302.88	302.13	301.19	300.02	299.65	298.60	297.94	296.76	295.83	295.81	295.78	295.79
	29	285.46	307.46	302.59	301.71	301.35	301.02	300.04	298.94	298.02	297.19	296.05	295.67	294.69	294.04	289.20	288.93	288.86	288.86	288.86
	30	296.84	318.41	314.47	313.38	313.08	312.78	309.87	309.17	306.87	305.92	304.69	304.29	303.30	302.77	301.76	301.14	301.08	301.07	301.07
E	13	279.61	301.49	297.04	296.83	296.05	295.47	294.83	294.40	293.81	293.05	292.01	291.67	290.59	289.83	285.08	284.06	284.00	284.00	284.00
	14	289.38	307.52	303.33	303.08	301.93	301.15	299.77	299.17	298.05	297.28	296.31	296.00	295.16	294.56	293.67	293.33	293.29	293.27	293.27
	15	287.22	309.71	304.67	303.96	303.67	303.37	302.43	301.82	301.00	300.17	299.08	298.67	297.48	296.69	291.67	291.05	291.00	290.99	290.99
H	22	318.26	342.74	338.33	337.23	336.84	336.49	335.47	334.98	333.99	333.13	331.97	331.49	330.31	329.53	324.24	323.39	323.32	323.31	323.32
	23	294.10	316.67	312.34	311.65	311.36	311.05	310.48	309.74	308.97	308.24	307.26	306.90	305.90	305.25	300.70	298.27	297.94	297.92	297.91
	24	293.57	317.40	313.17	312.46	312.19	311.87	311.39	310.73	309.94	308.78	307.49	307.09	305.97	305.03	298.75	298.40	298.36	298.34	298.34
I	25	286.72	309.17	305.22	304.34	304.00	303.72	302.80	302.17	301.57	300.67	299.40	298.96	297.74	296.87	291.33	290.99	290.93	290.93	290.96
	26	278.00	301.35	297.39	296.36	296.08	295.82	294.79	294.23	292.87	291.91	290.66	290.21	288.98	288.19	283.31	282.61	282.54	282.55	282.52
	27	287.38	309.26	304.32	303.33	302.87	302.44	299.63	298.56	294.79	293.34	292.18	291.85	291.85	---	---	---	---	---	291.85
D	10	280.56	303.39	299.33	298.82	298.20	297.74	296.80	296.14	294.85	293.75	292.36	291.91	290.49	289.31	287.21	285.16	284.96	284.94	284.94
	11	296.95	320.05	315.88	315.47	313.96	313.08	311.58	310.34	307.64	306.32	305.04	304.56	303.32	302.29	302.25	302.05	301.99	301.99	301.99
	12	292.98	315.24	311.50	311.71	310.05	309.26	307.99	306.85	305.65	304.60	303.26	302.77	301.44	300.64	297.50	296.82	296.77	296.75	296.75
L	34	292.39	316.46	311.51	310.73	310.33	309.73	308.66	307.68	305.82	304.80	303.47	303.01	301.69	300.83	298.15	296.86	296.80	296.79	296.79
	35	286.47	308.60	304.60	303.28	302.67	301.91	294.86	293.59	292.01	291.43	291.22	291.16	291.18	---	---	---	---	---	291.18
	36	278.12	303.83	299.19	298.02	297.42	296.60	285.94	283.72	283.16	283.16	283.22	283.10	283.13	---	---	---	---	---	283.13
K	31	287.98	310.51	306.08	305.11	304.83	304.54	303.77	303.20	301.94	301.04	299.77	299.29	298.04	297.26	292.85	292.31	292.27	292.24	292.23
	32	279.93	306.19	301.58	299.91	299.54	299.15	296.75	295.45	293.15	291.76	289.19	288.46	287.30	286.73	286.06	285.61	285.32	285.32	285.32
	33	295.49	318.18	313.57	312.49	312.03	311.54	309.01	308.04	306.80	305.68	304.19	303.68	302.38	301.46	300.20	300.02	299.95	299.96	299.96

Appendix G - Wet and Dry Cycle.

wet cycle 95-100% RH @ 25 °C dry cycle 25% RH @ 25 °C

sample no.	wt. of salt-impregnated sample (g)	wt. of sample following a 3-day wet cycle (g)	wt. of sample following a 3-day dry cycle (g)	wt. of sample following a 3-day wet cycle (g)	wt. of sample following a 3-day dry cycle (g)
16	304.04	316.10	315.85	326.52	325.46
17	287.41	296.79	296.23	307.79	306.27
18	291.31	301.63	301.31	309.74	309.26
19	300.60	314.37	314.25	319.45	319.74
20	288.86	299.65	299.60	305.22	305.12
21	300.16	309.18	308.91	319.58	319.09
28	295.88	305.99	305.22	314.09	312.23
29	288.96	300.51	299.17	304.65	304.44
30	301.17	312.06	311.45	321.20	320.35
13	284.05	291.93	291.55	298.75	298.53
14	293.33	304.56	304.38	308.98	309.05
15	291.04	303.54	303.31	310.48	310.00
22	323.40	335.16	334.55	344.72	343.86
23	297.99	309.68	309.41	315.52	315.46
24	298.44	311.03	310.77	315.39	315.05
25	291.04	300.43	299.94	307.71	307.14
26	282.64	294.74	294.01	302.68	301.82
27	292.16	301.39	300.63	309.98	309.11
10	285.01	296.15	295.86	303.59	303.19
11	302.07	313.76	313.49	322.34	321.52
12	296.82	306.97	306.84	316.96	316.98
34	296.90	309.52	308.53	319.10	318.49
35	291.35	299.69	298.17	304.72	304.17
36	283.38	293.68	291.90	303.97	301.04
31	292.33	300.75	299.98	306.83	306.54
32	285.36	297.92	296.47	304.43	301.93
33	300.05	313.75	312.04	317.42	317.41

Appendix H - Drying results for poultice #1.

* = slightly burned   ***** = completely burned. Burned samples were in an oven at approximately 160°C for an indeterminate time.

group	treatment type	degree burned	sample no.	wt. of beaker (g)	wt. of beaker + wet poultice (g)	wt. of wet Poultice (g)	hours dried at 65°C 24	48	72	96	120	144	168	192	216	240	264	288	312	wt of dry poultice (g)
B	Untreated Samples Poulticed with H2O	***	4	117.77	171.00	53.23	139.30	135.66	135.51	135.45	135.41	135.38	135.30	135.22	134.32	134.00	133.09	—	—	37.91
			5	80.03	131.30	51.27	108.76	100.62	96.89	96.71	96.71	—	—	—	—	—	—	—	—	34.59
			6	117.07	170.00	52.93	134.10	133.39	133.27	133.13	133.11	—	—	—	—	—	—	—	—	36.89
C	Untreated Samples Poulticed with Orvus		7	99.56	149.66	50.10	128.11	126.56	125.77	125.75	—	—	—	—	—	—	—	—	—	23.91
			8	99.76	149.43	49.67	127.82	125.83	125.37	125.34	—	—	—	—	—	—	—	—	—	24.09
			9	72.85	123.55	50.70	103.19	99.18	98.79	98.78	—	—	—	—	—	—	—	—	—	24.77
A	Untreated Samples Poulticed with Triton	**	1	182.98	245.92	62.94	219.47	219.41	219.19	219.17	219.52	219.07	214.92	—	—	—	—	—	—	31.00
		**	2	178.55	244.40	65.85	213.86	213.80	213.76	213.63	213.41	212.72	208.95	—	—	—	—	—	—	35.45
		**	3	177.92	232.47	54.55	209.80	209.69	209.50	209.06	209.16	208.86	205.47	—	—	—	—	—	—	27.00
F	Salt-impregnated Samples Poulticed with H2O	****	16	114.48	215.68	101.20	164.51	147.55	142.72	142.49	142.39	142.36	142.22	142.13	139.42	—	—	—	—	76.26
			17	117.12	217.11	99.99	173.60	160.33	145.57	144.30	144.28	—	—	—	—	—	—	—	—	72.83
			18	117.10	221.75	104.65	171.07	153.95	140.35	138.23	138.20	—	—	—	—	—	—	—	—	83.55
G	Salt-impregnated Samples Poulticed with Orvus	****	19	163.83	225.60	61.77	206.21	192.13	191.43	191.38	191.31	191.20	191.59	191.35	188.92	—	—	—	—	36.68
		***	20	130.00	199.05	69.05	176.71	164.86	164.67	164.60	164.47	163.84	163.40	162.00	161.91	—	—	—	—	37.14
		*	21	184.86	268.85	83.99	248.30	228.52	220.62	219.47	219.36	218.77	218.53	218.08	217.11	—	—	—	—	51.74
J	Salt-impregnated Samples Poulticed with Triton	*****	28	162.33	221.36	59.03	195.04	194.96	194.92	194.82	194.87	194.83	179.71	—	—	—	—	—	—	41.65
		****	29	191.70	261.29	69.59	230.12	229.98	229.52	229.50	229.42	229.33	224.74	—	—	—	—	—	—	36.55
		***	30	183.27	278.91	95.64	233.09	232.92	232.82	232.66	232.75	232.69	226.95	—	—	—	—	—	—	51.96
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O		13	98.56	170.74	72.18	131.03	123.76	123.26	123.07	123.05	—	—	—	—	—	—	—	—	47.69
			14	79.99	144.90	64.91	120.06	113.98	104.65	102.06	102.04	—	—	—	—	—	—	—	—	42.86
			15	49.02	129.02	80.00	101.36	94.32	85.29	75.92	75.88	75.85	75.80	75.74	75.43	75.49	75.16	74.87	74.53	54.49
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	***	22	103.03	161.82	58.79	146.06	133.26	127.88	127.79	127.70	127.60	127.90	127.60	124.52	—	—	—	—	37.30
		***	23	136.59	204.19	67.60	175.92	165.17	164.78	164.83	164.57	164.82	164.64	163.99	161.86	—	—	—	—	42.33
		*	24	136.60	201.51	64.91	173.41	164.03	163.71	163.69	163.54	163.32	163.29	163.26	161.14	—	—	—	—	40.37
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	**	25	136.55	212.22	75.67	181.68	169.66	169.49	169.43	169.40	169.36	169.31	169.29	165.71	—	—	—	—	46.51
		*	26	136.23	197.23	61.00	175.57	163.99	163.82	163.80	163.70	163.62	163.55	163.39	161.04	—	—	—	—	36.19
		***	27	139.08	199.71	60.63	182.36	167.32	166.68	166.65	166.63	166.51	166.36	166.27	163.48	—	—	—	—	36.23
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O		10	51.62	122.39	70.77	99.67	93.17	83.82	75.93	75.90	75.86	75.85	75.79	75.66	75.80	75.61	75.39	—	47.00
			11	51.59	126.81	75.22	97.21	90.00	81.42	77.74	77.67	77.60	77.59	77.54	77.31	77.49	77.21	77.15	—	49.66
			12	49.13	134.31	85.18	111.80	103.03	93.91	80.00	78.81	78.75	78.71	78.65	78.54	78.71	78.52	77.98	77.98	56.33
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus		34	38.73	105.23	66.50	70.83	66.45	66.06	65.88	65.52	65.64	65.13	64.87	64.87	—	—	—	—	40.36
			35	43.77	114.97	71.20	77.99	74.03	73.51	73.48	73.47	—	—	—	—	—	—	—	—	41.50
			36	51.84	113.04	61.20	100.74	92.59	86.11	78.01	77.93	77.82	77.76	77.93	77.67	77.55	76.82	—	—	36.22
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	***	31	217.77	300.33	82.56	253.49	253.29	253.12	252.97	253.29	252.91	248.83	—	—	—	—	—	—	51.50
		****	32	190.92	270.27	79.35	226.06	225.82	225.48	225.34	225.70	225.24	219.42	—	—	—	—	—	—	50.85
		*****	33	162.40	234.13	71.73	194.98	194.93	194.80	194.71	195.07	194.71	179.17	—	—	—	—	—	—	54.96

Appendix I - Drying results for poultice #2.

No samples burned during poultice #2

group	treatment type	sample no	wt of beaker (g)	wt of beaker + wet poultice (g)	wt of wet Poultice (g)	hours dried at 105°C							wt.of dry poultice (g)
						24	48	72	96	120	144	168	
B	Untreated Samples Poulticed with H2O	4	226.20	294.80	68.60	242.27	242.23	242.17	242.16	---	---	---	52.64
		5	99.47	181.71	82.24	115.14	114.91	114.90	114.85	114.85	---	---	66.86
		6	178.56	246.46	67.90	193.41	193.36	193.33	193.32	---	---	---	53.14
C	Untreated Samples Poulticed with Orvus	7	224.09	281.99	57.90	236.44	236.34	236.25	236.23	---	---	---	45.76
		8	224.61	292.94	68.33	235.94	235.81	235.78	235.78	---	---	---	57.16
		9	99.45	167.68	68.23	113.16	112.94	112.93	112.90	112.89	---	---	54.79
A	Untreated Samples Poulticed with Triton	1	99.54	184.63	85.09	119.76	119.56	119.54	119.53	119.52	---	---	65.11
		2	138.17	224.07	85.90	150.97	150.91	150.91	---	---	---	---	73.16
		3	191.76	267.32	75.56	209.14	209.12	209.08	209.08	---	---	---	58.24
F	Salt-impregnated Samples Poulticed with H2O	16	99.42	158.20	58.78	115.12	115.09	115.08	---	---	---	---	43.12
		17	137.06	211.09	74.03	151.19	151.02	150.96	150.96	---	---	---	60.13
		18	99.45	166.54	67.09	116.00	115.89	115.79	115.79	---	---	---	50.75
G	Salt-impregnated Samples Poulticed with Orvus	19	229.58	299.00	69.42	252.63	252.48	252.34	252.26	252.24	252.22	---	46.78
		20	40.91	101.05	60.14	57.31	57.21	57.10	57.08	57.08	---	---	43.97
		21	99.28	174.36	75.08	118.38	118.19	118.17	118.16	---	---	---	56.20
J	Salt-impregnated Samples Poulticed with Triton	28	38.71	99.74	61.03	52.41	52.35	52.31	52.28	52.28	---	---	47.46
		29	37.01	102.47	65.46	57.20	57.14	57.02	57.01	57.00	---	---	45.47
		30	217.78	287.28	69.50	241.06	240.92	240.87	240.82	240.79	240.76	240.74	46.54
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13	54.34	135.72	81.38	69.45	69.39	69.38	69.38	---	---	---	66.34
		14	21.66	97.66	76.00	36.12	36.07	36.01	36.01	---	---	---	61.65
		15	40.71	129.02	88.31	55.42	55.24	55.22	55.20	---	---	---	73.82
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22	174.70	261.51	86.81	188.32	188.25	188.23	188.20	188.21	---	---	73.30
		23	70.75	161.20	90.45	88.25	88.01	87.90	87.86	87.82	87.82	---	73.38
		24	21.84	99.04	77.20	36.72	36.64	36.59	36.56	36.55	---	---	62.49
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25	222.83	301.26	78.43	237.85	237.72	237.69	237.64	237.63	---	---	63.63
		26	54.73	139.77	85.04	70.49	70.38	70.37	70.36	---	---	---	69.41
		27	71.65	157.03	85.38	88.05	87.91	87.83	87.82	87.77	87.76	---	69.27
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10	223.72	299.73	76.01	238.59	238.56	238.54	238.52	238.52	---	---	61.21
		11	225.88	299.81	73.93	239.45	239.33	239.25	239.25	---	---	---	60.56
		12	21.69	104.12	82.43	39.34	39.21	39.20	39.18	39.19	---	---	64.93
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34	139.63	212.28	72.65	154.55	154.49	154.40	154.36	154.33	154.34	---	57.94
		35	21.75	97.56	75.81	36.43	36.37	36.30	36.28	36.28	---	---	61.28
		36	43.68	113.01	69.33	59.88	59.76	59.65	59.65	---	---	---	53.36
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31	190.86	273.03	82.17	205.52	205.45	205.39	205.38	---	---	---	67.65
		32	192.00	259.88	67.88	207.31	207.23	207.21	207.19	207.20	---	---	52.68
		33	44.44	104.65	60.21	61.27	61.12	61.09	61.05	61.05	---	---	43.60

Appendix J - Conductimetric measurements and amount NaCl extracted forpoultice #1.

* = slightly burned ***** = completely burned. *K* for Tap H2O = 330 μS, Orvus = 50 μS, and Triton = 75 μS. M.W. NaCl = 58.44. Molar concentration ***(x) = [y-b] / m*** (avg), where the average slope value ***(m) = 97926.2*** , ***b = 0*** , and ***y =*** *measured conductance* ***(K)*** Therefore, molar concentration ***(x) = K / m*** (avg).

				A	B	C	D	E	F	G	H	I	J	K	L	M	N
group	treatment type	degree burned	sample no	amount of H2O added (L)	K (μS)	50 ml diluted to 100 ml	K (μS)	calculated molar concentration (mol/L) where x = K / m avg	NaCl poulticed (g/L) = x (mol/L) * M.W. NaCl (g/mol)	wt. of NaCl (g) [F * A]	NaCl from tap H2O (g)	NaCl from respective solutions (g)	wt. of NaCl (g) [H + I]	wt. of NaCl (g) [G - J]	NaCl poulticed (% w/w) [K - dry wt. of sample]	avg. amount of NaCl poulticed for each group (%w/w)	stdv (±)
B	Untreated Samples Poulticed with H2O	•••	4	0.4	5920	no	—	0.06045	3.53291	1.41317	0.079	0.079	0.158	1.25562	0.41	0.23	0.15
			5	0.4	1200	no	—	0.01225	0.71613	0.28645				0.12890	0.05		
			6	0.4	3480	no	—	0.03554	2.07678	0.83071				0.67316	0.23		
C	Untreated Samples Poulticed with Orvus		7	0.4	2040	no	—	0.02083	1.21742	0.48697	0.079	0.012	0.091	0.39626	0.13	0.11	0.03
			8	0.4	1230	no	—	0.01256	0.73403	0.29361				0.20290	0.07		
			9	0.4	2010	no	—	0.02053	1.19952	0.47981				0.38910	0.14		
A	Untreated Samples Poulticed with Triton	••	1	0.4	4190	no	—	0.04279	2.50049	1.00020	0.079	0.018	0.097	0.90352	0.31	0.32	0.01
		••	2	0.4	4130	no	—	0.04217	2.46468	0.98587				0.88919	0.31		
		••	3	0.4	4250	no	—	0.04340	2.53630	1.01452				0.91784	0.33		
E	Salt-impregnated Samples Poulticed with H2O	••••	16	0.4	>20000	yes	12620	0.25775	15.06262	6.02505	0.079	0.079	0.158	5.86750	1.958	1.67	0.39
			17	0.4	13900	no	—	0.14194	8.29519	3.31807				3.16052	1.119		
			18	0.4	>20000	yes	11820	0.24141	14.10778	5.64311				5.48556	1.921		
G	Salt-impregnated Samples Poulticed with Orvus	••••	19	0.4	>20000	yes	13210	0.26980	15.76682	6.30673	0.079	0.012	0.091	6.21602	2.063	1.72	0.31
		•••	20	0.4	16050	no	—	0.16390	9.57825	3.83130				3.74059	1.318		
		•	21	0.4	>20000	yes	11200	0.22874	13.36778	5.34711				5.25640	1.778		
J	Salt-impregnated Samples Poulticed with Triton	•••••	28	0.4	16850	no	—	0.17207	10.05567	4.02227	0.079	0.018	0.097	3.92559	1.346	1.75	0.43
		••••	29	0.4	19040	no	—	0.19443	11.36261	4.54505				4.44837	1.558		
		•••	30	0.4	>20000	yes	14820	0.30268	17.68844	7.07538				6.97870	2.351		
F	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O		13	0.4	10380	no	—	0.10600	6.19453	2.47781	0.079	0.079	0.158	2.32026	0.830	1.11	0.32
			14	0.4	12110	no	—	0.12366	7.22696	2.89078				2.73323	0.945		
			15	0.4	19290	no	—	0.19699	11.51181	4.60472				4.44717	1.548		
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	•••	22	0.4	17650	no	—	0.18024	10.53310	4.21324	0.079	0.012	0.091	4.12253	1.295	1.41	0.09
		•••	23	0.4	19010	no	—	0.19413	11.34471	4.53788				4.44717	1.512		
		•	24	0.4	17720	no	—	0.18095	10.57487	4.22995				4.13924	1.410		
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	••	25	0.4	17960	no	—	0.18340	10.71810	4.28724	0.079	0.018	0.097	4.19056	1.462	1.48	0.02
		•	26	0.4	17780	no	—	0.18157	10.61068	4.24427				4.14759	1.492		
		•••	27	0.4	18450	no	—	0.18841	11.01052	4.40421				4.30753	1.499		
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O		10	0.4	>20000	yes	11280	0.23038	13.46327	5.38531	0.079	0.079	0.158	5.22776	1.863	1.82	0.09
			11	0.4	>20000	yes	10840	0.22139	12.93810	5.17524				5.01769	1.690		
			12	0.4	>20000	yes	12040	0.24590	14.37036	5.74815				5.59060	1.908		
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus		34	0.4	>20000	yes	10860	0.22180	12.96197	5.18479	0.079	0.012	0.091	5.09408	1.742	1.31	0.40
			35	0.4	9690	no	—	0.09895	5.78276	2.31310				2.22239	0.776		
			36	0.4	16820	no	—	0.17176	10.03777	4.01511				3.92440	1.411		
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	•••	31	0.4	17620	no	—	0.17993	10.51519	4.20608	0.079	0.018	0.097	4.10940	1.427	1.37	0.13
		••••	32	0.4	17920	no	—	0.18299	10.69422	4.27769				4.18101	1.494		
		•••••	33	0.4	15200	no	—	0.15522	9.07099	3.62840				3.53172	1.195		

Appendix K - Amount NaCl extracted as indicated by titrator strips fopoultice #1.

* = slightly burned ***** = completely burned. **Wt. of salt poulticed (g)** = NaCl (g/mL) * amount of water added (mL) * diluted volume (mL) / measured sample solution (mL). **NaCl poulticed (%w/w)**= wt. of salt poulticed (g) / wt. of dry sample.

group	treatment type	degree burned	sample no	amount of H_2O added (mL)	Quantab			50 mL diluted to 100 mL	Quantab			50 mL diluted to 100 mL	Quantab			NaCl poulticed (g)	NaCl poulticed (% w/w)	avg amount of NaCl poulticed for each group (%w/w)	stdv (±)
					scale	NaCl (g/mL)	Cl- (g/mL)		scale	NaCl (g/mL)	Cl- (g/mL)		scale	NaCl (g/mL)	Cl- (g/mL)				
B	Untreated Samples Poulticed with H_2O	***	4	400	1.8	0.00066	0.00040	no	—	—	—	no	—	—	—	0.264	0.09	0.124	0.037
			5	400	0.4	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—		
			6	400	2.6	0.00116	0.00066	no	—	—	—	no	—	—	—	0.464	0.16		
C	Untreated Samples Poulticed with Orvus		7	400	1.0	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—	0.078	0.000
			8	400	1.0	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—		
			9	400	1.6	0.00056	0.00032	no	—	—	—	no	—	—	—	0.224	0.08		
A	Untreated Samples Poulticed with Triton	**	1	400	0.4	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—	0.000	0.000
		**	2	400	0.4	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—		
		**	3	400	0.4	< 0.00046	< 0.000282	no	—	—	—	no	—	—	—	—	—		
F	Salt-impregnated Samples Poulticed with H_2O	****	16	400	9.4	> 0.01036	> 0.006185	yes	6.8	0.00730	0.00443	no	—	—	—	5.840	1.949	1.604	0.352
			17	400	7.0	0.00792	0.00475	no	—	—	—	no	—	—	—	3.168	1.121		
			18	400	8.8	> 0.01036	> 0.006284	yes	6.4	0.00622	0.00377	no	—	—	—	4.976	1.742		
G	Salt-impregnated Samples Poulticed with Orvus	****	19	400	9.0	> 0.01036	> 0.006284	yes	6.6	0.00673	0.00408	no	—	—	—	5.384	1.787	1.553	0.261
		***	20	400	8.4	> 0.01036	> 0.006284	yes	5.4	0.00422	0.00256	no	—	—	—	3.376	1.189		
		*	21	400	8.6	> 0.01036	> 0.006284	yes	6.4	0.00622	0.00377	no	—	—	—	4.976	1.683		
J	Salt-impregnated Samples Poulticed with Triton	*****	28	400	7.6	0.01036	0.00628	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.251	1.492	0.321
		****	29	400	9.2	> 0.01036	> 0.006284	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.278		
		***	30	400	9.4	> 0.01036	> 0.006284	yes	7.8	> 0.01036	> 0.006284	yes	5.0	0.00361	0.00219	5.776	1.946		
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H_2O		13	400	5.4	0.00422	0.00256	no	—	—	—	no	—	—	—	1.688	0.604	0.923	0.398
			14	400	5.8	0.00493	0.00299	no	—	—	—	no	—	—	—	1.972	0.681		
			15	400	7.8	> 0.01036	> 0.006284	yes	6.0	0.00533	0.00323	no	—	—	—	4.264	1.485		
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	***	22	400	8.8	> 0.01036	> 0.006284	yes	5.8	0.00493	0.00299	no	—	—	—	3.944	1.239	1.274	0.047
		***	23	400	8.4	> 0.01036	> 0.006284	yes	5.8	0.00493	0.00299	no	—	—	—	3.944	1.341		
		*	24	400	7.8	> 0.01036	> 0.006284	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.243		
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	**	25	400	7.8	> 0.01036	> 0.006284	yes	5.4	0.00422	0.00256	no	—	—	—	3.376	1.177	1.253	0.056
		*	26	400	7.8	> 0.01036	> 0.006284	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.312		
		***	27	400	9.0	> 0.01036	> 0.006284	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.269		
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H_2O		10	400	8.8	> 0.01036	> 0.006284	yes	6.6	0.00673	0.00408	no	—	—	—	5.384	1.919	1.722	0.152
			11	400	8.4	> 0.01036	> 0.006284	yes	6.2	0.00575	0.00349	no	—	—	—	4.600	1.549		
			12	400	8.6	> 0.01036	> 0.006284	yes	6.4	0.00622	0.00377	no	—	—	—	4.976	1.698		
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus		34	400	8.2	> 0.01036	> 0.006284	yes	6.0	0.00533	0.00323	no	—	—	—	4.264	1.458	1.034	0.500
			35	400	4.0	0.00237	0.00144	no	—	—	—	no	—	—	—	0.948	0.331		
			36	400	8.2	> 0.01036	> 0.006284	yes	5.6	0.00456	0.00277	no	—	—	—	3.648	1.312		
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	***	31	400	7.8	> 0.01036	> 0.006284	yes	5.4	0.00422	0.00256	no	—	—	—	3.376	1.172	1.150	0.057
		****	32	400	7.8	> 0.01036	> 0.006284	yes	5.4	0.00422	0.00256	no	—	—	—	3.376	1.206		
		*****	33	400	7.0	0.00792	0.00481	no	—	—	—	no	—	—	—	3.168	1.072		

Appendix L- Conductimetric measurements and amount NaCl extracted for poultice #2.

M.W. NaCl = 58.44. Molar concentration $(x) = [y-b] / m$ (avg), where the average slope value $(m) = 97926.2$, $b = 0$, and $y =$ *measured conductance* (K) Therefore, molar concentration $(x) = K / m$ (avg).

			A	B	C	D	E	F	H	I	J
group	treatment type	sample no	amount of H2O added (L)	K (µS)	50 mL diluted to 100 mL	calculated molar concentration (mol/L) where x = K / m avg	NaCl poulticed (g/L) = x (mol/L)* *M.W. NaCl* (g/mol)	wt. of NaCl (g) [F * A]	NaCl poulticed (% w/w) [K - dry wt. of sample]	avg. amount of NaCl poulticed for each group [%w/w]	stdv (±)
B	Untreated Samples Poulticed with H2O	4	0.4	4190	no	0.04279	2.50049	1.00020	0.33	0.38	0.04
		5	0.4	4470	no	0.04565	2.66759	1.06704	0.39		
		6	0.4	4980	no	0.05085	2.97194	1.18878	0.41		
C	Untreated Samples Poulticed with Orvus	7	0.4	4820	no	0.04922	2.87646	1.15058	0.38	0.47	0.10
		8	0.4	5140	no	0.05249	3.06743	1.22697	0.42		
		9	0.4	7190	no	0.07342	4.29082	1.71633	0.60		
A	Untreated Samples Poulticed with Triton	1	0.4	4000	no	0.04085	2.38710	0.95484	0.33	0.33	0.01
		2	0.4	3980	no	0.04064	2.37517	0.95007	0.33		
		3	0.4	3710	no	0.03789	2.21404	0.88562	0.32		
F	Salt-impregnated Samples Poulticed with H2O	16	0.4	9180	no	0.09374	5.47840	2.19136	0.73	0.76	0.02
		17	0.4	8920	no	0.09109	5.32324	2.12930	0.75		
		18	0.4	9430	no	0.09630	5.62760	2.25104	0.79		
G	Salt-impregnated Samples Poulticed with Orvus	19	0.4	15390	no	0.15716	9.18438	3.67375	1.22	1.07	0.19
		20	0.4	9470	no	0.09671	5.65147	2.26059	0.80		
		21	0.4	14750	no	0.15062	8.80245	3.52098	1.19		
J	Salt-impregnated Samples Poulticed with Triton	28	0.4	10890	no	0.11121	6.49889	2.59956	0.89	1.09	0.14
		29	0.4	14350	no	0.14654	8.56373	3.42549	1.20		
		30	0.4	14670	no	0.14981	8.75470	3.50188	1.18		
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13	0.4	9160	no	0.09354	5.46647	2.18659	0.78	0.77	0.05
		14	0.4	8540	no	0.08721	5.09647	2.03859	0.70		
		15	0.4	10000	no	0.10212	5.96776	2.38710	0.83		
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22	0.4	10300	no	0.10518	6.14679	2.45872	0.77	0.85	0.12
		23	0.4	12640	no	0.12908	7.54325	3.01730	1.03		
		24	0.4	9410	no	0.09609	5.61566	2.24626	0.77		
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25	0.4	9610	no	0.09814	5.73502	2.29401	0.80	0.93	0.09
		26	0.4	11500	no	0.11744	6.86292	2.74517	0.99		
		27	0.4	11970	no	0.12223	7.14341	2.85736	0.99		
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10	0.4	9440	no	0.09640	5.63356	2.25343	0.80	0.89	0.24
		11	0.4	8110	no	0.08282	4.83985	1.93594	0.65		
		12	0.4	15070	no	0.15389	8.99341	3.59737	1.23		
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34	0.4	11400	no	0.11641	6.80325	2.72130	0.93	0.90	0.03
		35	0.4	10290	no	0.10508	6.14082	2.45633	0.86		
		36	0.4	10740	no	0.10967	6.40937	2.56375	0.92		
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31	0.4	8580	no	0.08762	5.12034	2.04814	0.71	0.82	0.11
		32	0.4	8960	no	0.09150	5.34711	2.13884	0.76		
		33	0.4	12040	no	0.12295	7.18518	2.87407	0.97		

Appendix M - Amount NaCl extracted as indicated by titrator strips for poultice #2.

* = slightly burned ***** = completely burned. **Wt. of salt poulticed (g)** = NaCl (g/mL) * amount of water added (mL) * diluted volume (mL) / measured sample solution (mL). **NaCl poulticed (%w/w)** = wt. of salt poulticed (g) / wt. of dry sample.

group	treatment type	sample no.	amount of H2O added (mL)	Quantab			50 mL diluted to 100 mL	NaCl poulticed (g)	NaCl poulticed (% w/w)	avg. amount of NaCl poulticed for each group (%w/w)	stdv (±)
				scale	NaCl (g/mL)	Cl- (g/mL)					
B	Untreated Samples Poulticed with H2O	4	400	1.2	< 0.00046	< 0.000282	no	—	—	0.092	0.000
		5	400	1.2	< 0.00046	< 0.000282	no	—	—		
		6	400	1.8	0.00066	0.00040	no	0.264	0.09		
C	Untreated Samples Poulticed with Orvus	7	400	2.4	0.00102	0.00062	no	0.408	—	0.276	0.000
		8	400	2.4	0.00102	0.00062	no	0.408	—		
		9	400	3.6	0.00197	0.00120	no	0.788	0.28		
A	Untreated Samples Poulticed with Triton	1	400	0.8	< 0.00046	< 0.000282	no	—	—	0.000	0.000
		2	400	0.4	< 0.00046	< 0.000282	no	—	—		
		3	400	0.4	< 0.00046	< 0.000282	no	—	—		
F	Salt-impregnated Samples Poulticed with H2O	16	400	5.0	0.00361	0.00219	no	1.444	0.482	0.437	0.034
		17	400	4.4	0.00282	0.00171	no	1.128	0.399		
		18	400	4.6	0.00307	0.00186	no	1.228	0.430		
G	Salt-impregnated Samples Poulticed with Orvus	19	400	7.0	0.00792	0.00481	no	3.168	1.051	0.945	0.322
		20	400	5.0	0.00361	0.00219	no	1.444	0.509		
		21	400	7.4	0.00943	0.00572	no	3.772	1.276		
J	Salt-impregnated Samples Poulticed with Triton	28	400	6.0	0.00533	0.00323	no	2.132	0.731	1.115	0.296
		29	400	7.6	0.01036	0.00628	no	4.144	1.452		
		30	400	7.2	0.00863	0.00523	no	3.452	1.163		
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13	400	4.8	0.00333	0.00202	no	1.332	0.476	0.482	0.049
		14	400	4.6	0.00307	0.00186	no	1.228	0.424		
		15	400	5.2	0.00391	0.00237	no	1.564	0.545		
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22	400	5.6	0.00456	0.00277	no	1.824	0.573	0.651	0.139
		23	400	6.4	0.00622	0.00377	no	2.488	0.846		
		24	400	5.2	0.00391	0.00237	no	1.564	0.533		
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25	400	5.0	0.00361	0.00219	no	1.444	0.504	0.691	0.137
		26	400	6.2	0.00575	0.00349	no	2.300	0.827		
		27	400	6.0	0.00533	0.00323	no	2.132	0.742		
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10	400	5.0	0.00361	0.00219	no	1.444	0.515	0.739	0.390
		11	400	4.6	0.00307	0.00186	no	1.228	0.414		
		12	400	7.4	0.00943	0.00572	no	3.772	1.287		
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34	400	5.8	0.00493	0.00299	no	1.972	0.674	0.595	0.070
		35	400	5.0	0.00361	0.00219	no	1.444	0.504		
		36	400	5.4	0.00422	0.00256	no	1.688	0.607		
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31	400	4.6	0.00307	0.00186	no	1.228	0.426	0.523	0.104
		32	400	4.8	0.00333	0.00202	no	1.332	0.476		
		33	400	5.8	0.00493	0.00299	no	1.972	0.667		

Appendix N - Desalination results: A summary.

			A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P
group	treatment type	sample no.	average NaCl originally in stone (%w/w)	average NaCl introduced (% w/w)	stdv. (±)	total NaCl in stone (%w/w) [A+C]	average NaCl obtained from the 1st round of poulticing "Pocket Pal" (% w/w)	stdv. (±)	average NaCl obtained from the 1st round of poulticing "Quantab" (% w/w)	stdv. (±)	average NaCl obtained from the 2nd round of poulticing according to "Pocket Pal" (% w/w)	stdv. (±)	average NaCl obtained from the 2nd round of poulticing according to "Quantab" (% w/w)	stdv. (±)	total NaCl poulticed "Pocket Pal" (%w/w) [E+I]	% NaCl removed according to "Pocket Pal" [(M/D)*100]	total NaCl poulticed "Quantab" (%w/w) [G+K]	% NaCl removed according to "Quantab" [(O/D)*100]
B	Untreated Samples Poulticed with H2O	4 5 6	2.47	—	---	2.47	0.23	0.15	0.12	0.04	0.36	0.03	0.09	0.00	0.59	23.85	0.22	8.74
C	Untreated Samples Poulticed with Orvus	7 8 9	2.47	---	—	2.47	0.11	0.03	0.08	0.00	0.45	0.08	0.28	0.00	0.56	22.71	0.35	14.33
A	Untreated Samples Poulticed with Triton	1 2 3	2.47	---	—	2.47	0.32	0.01	0.00	0.00	0.31	0.01	0.00	0.00	0.62	25.22	0.00	0.00
F	Salt-impregnated Samples Poulticed with H2O	16 17 18	2.47	1.72	0.22	4.19	1.67	0.39	1.60	0.35	0.72	0.02	0.44	0.03	2.39	56.97	2.04	48.71
G	Salt-impregnated Samples Poulticed with Orvus	19 20 21	2.47	1.57	0.08	4.04	1.72	0.31	1.55	0.26	1.04	0.21	0.95	0.32	2.76	68.25	2.50	61.86
J	Salt-impregnated Samples Poulticed with Triton	28 29 30	2.47	1.36	0.11	3.83	1.75	0.43	1.49	0.32	1.05	0.13	1.12	0.30	2.80	73.07	2.61	68.10
E	Salt-impregnated Samples Brushed with Orvus, Poulticed with H2O	13 14 15	2.47	1.41	0.12	3.88	1.11	0.32	0.92	0.40	0.73	0.05	0.48	0.05	1.83	47.25	1.41	36.22
H	Salt-impregnated Samples Brushed with Orvus, Poulticed with Orvus	22 23 24	2.47	1.50	0.15	3.97	1.41	0.09	1.27	0.05	0.84	0.10	0.65	0.14	2.25	56.63	1.93	48.45
I	Salt-impregnated Samples Brushed with Orvus, Poulticed with Triton	25 26 27	2.47	1.55	0.06	4.02	1.48	0.02	1.25	0.06	0.87	0.08	0.69	0.14	2.35	58.41	1.94	48.32
D	Salt-impregnated Samples Brushed with Triton, Poulticed with H2O	10 11 12	2.47	1.52	0.17	3.99	1.82	0.09	1.72	0.15	0.85	0.24	0.74	0.39	2.67	67.10	2.46	61.76
L	Salt-impregnated Samples Brushed with Triton, Poulticed with Orvus	34 35 36	2.47	1.65	0.12	4.12	1.31	0.40	1.03	0.50	0.85	0.04	0.60	0.07	2.16	52.40	1.63	39.54
K	Salt-impregnated Samples Brushed with Triton, Poulticed with Triton	31 32 33	2.47	1.64	0.20	4.11	1.37	0.13	1.15	0.06	0.78	0.12	0.52	0.10	2.15	52.26	1.67	40.73

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INDEX