THE EFFECT OF HERBICIDE ON STONE AND MASONRY MATERIAL

by

CAITLIN MAREDITH OSHIDA

(Under the Direction of John Waters)

ABSTRACT

This study examined the effects of herbicides on stone and masonry materials. Although the ecological effects of herbicides are well studied, it is unclear how herbicides affect the stone and masonry material that they contact. This experiment tested Roundup® and Garlon®4, on brick, limestone, concrete, and granite. After exposure to the herbicides, the stone and masonry samples were placed in a QUV to undergo artificial weathering. The samples were monitored periodically for physical and chemical changes by measuring these parameters weight and pH, and using the following tests: colorimetry, Fouier transform infrared spectroscopy, absorption, and X-ray defraction. The results showed that these herbicides negatively affected the materials in the following ways: salt formation and color change. While Garlon®4 changes the aesthetics of the material, Roundup®can lead to a long-term, increased rate of deterioration.

INDEX WORDS: Herbicide, Roundup®, Garlon®4, Masonry material, Brick, Concrete, Limestone, Granite, QUV, National Park Service, National Center for Preservation Technology and Training

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DEDICATION

For my family and friends who have always encouraged and supported me.

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My ability to complete this thesis has been dependent on the help and guidance of numerous people. Mary Streigel and Debbie Smith at the National Center for Preservation Technology and Training (NCPTT) who provided me with the opportunity to have the lead on this project and encouraged my work on it as my thesis topic. To Jason Church, whose invaluable assistance supported my endeavor to carry out this experiment at NCPTT while attending school at The University of Georgia. I am grateful for your time, knowledge, support and friendship throughout this process. I offer a big "Thank you," to my major professor John Waters, who encouraged and welcomed the chance for an environmental science-based thesis to be produced for the first time in the Historic Preservation Program. Hopefully, this will be a step toward a closer relationship between environmental science and historic preservation. To my thesis reading committee, Dr. Wayde Brown, Dr. Mary Striegel, and Dr. Timothy Dore, I am grateful for your time, consideration, and insightful suggestions.

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AUTHOR'S NOTE

In the summer of 2010, I was an intern at the U.S. National Park Service's National Center for Preservation Technology and Training (NCPTT) in Natchitoches, Louisiana (Figure 0.1). This position called for a person with a scientific background and a knowledge and passion for historical features and objects. It was a perfect fit for me, as a recent graduate with a B.S. in Environmental Science and a B.A. in Historic Preservation from the University of Mary Washington. At NCPTT, I combined the two fields, applying science to real world historic preservation situations.

Figure 0.1. National Center for Preservation Technology and Training, Natchitoches, Louisiana. "NCPTT Offices are Located at Lee H." *NCPTT Offices*. Web. 23 Aug 2011.

My job for the summer was to research and develop an experiment that could be conducted (the following summer by another intern) that would test for the effects that an herbicide had, if any, on stone and masonry material. I spent two months researching, recording, surveying, and developing an experiment that would do just that. Then, I departed for the University of Georgia (UGA) to begin my Master's Degree Program in

Historic Preservation (MHP). The project I had generally designed, but not yet refined, was left to be carried out by the next intern who received the position at NCPTT. When I arrived at UGA, I immediately told Professor John Waters about my herbicide project and how such a study would benefit the historic preservation field, especially the U.S National Park Service. He listened to my ideas and the status of the study design and agreed to let me refine and implement my experimental design that I had started at NCPTT, with the final product being this thesis. A Memorandum of Understanding was signed between NCPTT and UGA, and the research design I had started at NCPTT was mine once again to refine and experiments mine to conduct.

During the past year, I have traveled multiple times to Natchitoches to complete different stages of the experiment. There have been numerous hurdles and many phones calls to NCPTT for clarification, more information, and data analysis guidance. The study is finally complete.

In an effort to marry environmental science with historic preservation, and finish what I started in June of 2010, I give you: The Effects of Herbicide on Stone and Masonry Materials, as a thesis to satisfy requirements for the Master of Historic Preservation degree.

CHAPTER 1

INTRODUCTION

Purpose of the Study

Despite the upmost care and attention, unwanted ivy vines, blades of grass, and other invasive vegetation can creep onto, on, or near historic features. They can take root in cracks and crevices, which can cause damage to the material as they grow and expand, leading to accelerated deterioration of the historic materials. These plants can be unsightly, and, when left unchecked, can detract from the historic fabric of the site. So how is this problem dealt with? More often than not herbicides are implemented to combat the unwanted vegetation in the area. They are applied with a single goal: to kill the plants. But what about the historic features?

Although the ecological effects of herbicides are well known and documented (by both manufacturing companies and the Environmental Protection Agency), it is unclear how herbicides affect the stone and masonry material that they contact. The purpose of this study is to determine the effects herbicides have on stone and masonry materials. The Materials Research Program and Historic Landscapes Programs at the National Center for Preservation Technology and Training (NCPTT) and the University of Georgia (UGA), respectively, have jointly given the author permission and encouragement to develop, design, and conduct research that will test the effects of herbicides on historic stone and masonry in order to better understand how to preserve our nation's historic features.

Vegetation increases the rate of masonry deterioration as roots grow into and around structures (Figure 1.1). The growing roots can expand within and against the surfaces of structures causing further cracking, which can make the material more vulnerable to moisture. Water and chemicals from herbicides applied to or near structures can enter the material through absorption (capillary effect) and spread throughout the material, causing deterioration from the inside-out. Most herbicides are salt based. When salt-based herbicides are applied, this can lead to salt crystallization (efflorescence) occurring on and within the material. Efflorescence development can increase deterioration of the material through its hydration cycle (the expansion and contraction within cracks as it becomes wet and liquefies then dries and solidifies) causing the cracks to widen.

Figure 1.1. Fort Wadsworth, Staten Island, New York, Gateway National Recreation Park.
8 May 2007. Inside the fort; example of vegetation growing in and around historic sites.
Photo courtesy of Kerry Mitchell.

From an historic preservation perspective, the protection and conservation of the nation's historic buildings, features, monuments, cemeteries, and other structures are of upmost importance. It is recognized that as these features age they will naturally deteriorate due to stress from the weather and environment. However, degradation and deterioration will also occur through man-made impacts, such as weed-eaters, human weight (floors of structures), construction, and many other examples. Chemicals that are introduced into the environment, through aerosols, insecticides, pesticides, and herbicides might not only harm the natural environment, but could damage stone and masonry structures. The chemicals could accelerate deterioration rates by altering the chemical bonds of the masonry structure, thereby making them weaker and less able to support themselves and the rest of the structure, possibly leading to instability and eventually failure.

It is argued by many within the property management field that their employees make sure not to spray or apply herbicide to any historic structures on their property. Therefore, this study does not apply to them because it is impossible that herbicide could be contacting their historic masonry, and therefore herbicides are not affecting deterioration rates. Many of these well-meaning people would be mistaken. Even if a person does not spray or apply herbicide directly on or around historic structures, the wind is capable of carrying the herbicides to nearby structures and coating them with the chemicals. In a test conducted by Jason Church, NCPTT, dye was added to water and applied to plants near structures. This was done to provide evidence and an example of how far liquids could spread and where they could potentially end up[1]. This demonstrates that even though people take proper care in trying to keep herbicides off of

[1] Church, Jason. NPS National Preservation of Technology and Training. 2010.

historic features, there is no guarantee that the herbicide will go exactly where it is aimed or applied, and only there.

This experiment was designed to determine exactly what kind of affects the herbicides Roundup® and Garlon®4 have on brick, limestone, concrete, and granite materials. This information should inform decision makers whether precautions must be taken before herbicides are used on or near historic masonry materials.

Expected Results

This study was prompted by growing concern within the U.S. National Park Service on the use of herbicides on and around historic features, and the overall lack of information available on this subject. The major evidence to support this concern was found in two theses written in 1989 and 1999. The first one, Linda Anne Cook's thesis, "The Effects of Herbicides on Masonry: Products, Choices and Testing[2]," tested the use of three herbicides (Roundup®, Weed-B-Gon®, and borax) on three types of building material (brick, limestone, and mortar). Cook completely submerged the materials for 14 to 16 hours and let them dry for 4 to 5 hours (one cycle). She performed this cycle 10 times. Cook concluded that there was minor surface pitting and imbedded salt crystals, as well as discoloration in all of the test samples at the end of the experiment. In 1999, Catherine Camille Dewey's thesis, "An Investigation into the Effects of an Herbicide on Historic Masonry Materials[3]," explored the use of different concentrations of the herbicide Roundup® on brick, sandstone, and limestone. Again, Dewey's test involved complete immersion for 24 hours and a dry period of 24 hours (one cycle). This was

[2] Cook, Linda Anne. "The Effects of Herbicides on Masonry: Products. Choices, and Testing." Thesis, Columbia University, 1989.
[3] Dewey, Catherine Camille. "An Investigation Into the Effects of an Herbicide on Historic Masonry Material." Thesis, University of Pennsylvania, 1999.

performed on the samples through seven to ten cycles. Like Cook, Dewey concluded that there were negative effects on the materials. However, both of these tests were unrealistic as all masonry materials exposed to herbicide were submerged for hours in the solution. This research will update, better quantitate, and expand upon Dewey's and Cook's information using more advanced technology to determine the effects of two of today's most popular herbicides on stone and masonry.

Based on previous studies performed by Cook and Dewey, herbicides have negative effects on stone and masonry, such as pitting and efflorescence. Therefore, in this study, it is expected that the herbicides will cause negative attributes to the stone and masonry. Because herbicides are salt based (to help kill vegetation by depriving them of water through osmosis), efflorescence should develop on the surface of the exposed masonry materials, and due to interactions between the herbicides and masonry and stone, pitting is likely to occur. Cook identified areas of pitting, which could be attributed to the deterioration of cellular bonds through chemical interactions. Pitting will be monitored on the surface of the samples within this study. These chemical bonds also hold the material together, and alterations of the chemical structure could affect the material's hardness and durability, making it more porous, weaker, and more prone to failure. Therefore, the hardness of the materials will be tested. Some herbicides contain dyes to make it easier to see where they have been applied. Dyes can cause color changes to materials over time permanently staining them, and changing the historic fabric and character of the structure. Although not a form of physical deterioration of the material, such as cracking and pitting, changes in the color destroy the historic integrity of the feature and cause man-made imperfections in the feature. Stains can be eye-sores, detract from the overall experience of seeing the historic feature, and make the feature look in worse condition than it actually is (Figure 1.2).

Figure 1.2. Applying Roundup® herbicide to unwanted vegetation near a masonry structure.

This study on the effects of herbicides on stone and masonry will provide information toward the protection of historic structures from manmade landscaping aides. Unwanted vegetation is a constant pest to those working to protect historic sites, and often, herbicides are a quick and easy way to get rid of the problem. The general effects that these chemicals have on the environment have been documented, however, the effects they have on stone and masonry structures have not. This study seeks to provide qualitative and quantitative information concerning the impact of exposing historic features made of stone and masonry material to herbicides.

CHAPTER 2

BACKGROUND INFORMATION: HERBICIDES, MASONRY MATERIALS, AND SALT

Weeds and unwanted vegetation have negatively impacted human efforts at cultivation for centuries. Over time many methods of removal have been developed from the humble "hand-weeding to include primitive hoes (6000 B.C.), animal-powered implements (1000 A.D.), mechanically powered implements (1920 A.D.), biological control (1930 A.D.), and chemical (herbicide) control (1947 A.D.)."[4] Since the production of the "first selective herbicides, 2, 4-D and MCPA, in 1947, herbicides have had a major positive impact on world agricultural production."[5]

Herbicides are often the most reliable and the least expensive method of weed control available to the public. However, over time, plants and vegetation can build up a resistance to the chemicals and become more and more difficult to get rid of. This leads to newer, stronger chemical solutions being developed to overcome this newfound resistance. Rachel Carson's book, Silent Spring, written in 1962, facilitated the ban on dichlorodiphenyltrichloroethane (DDT) pesticide and started an environmental movement in the United States[6]. In many cases this has also led to environmental studies conducted by the Environmental Protection Agency (EPA) on herbicides to determine the impact on the ecological system in which the chemicals are coming into contact. However, today

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[4] Powles, Stephen B., and Dale L. Shaner. *Herbicide Resistance and World Grains*. Boca Raton: CRC Press LLC, 2001. 1. Print.
[5] Ibid.
[6] Carson, Rachel. *Silent Spring*. Boston: Houghtib Mifflin Company, 1962. Print.

herbicides are used by the National Park Service and the public extensively across the nation to rid crops, lawns, buildings, and other structures of unwanted vegetation growth.

Table 2.1 shows the most commonly used herbicides in the United States. The two herbicides used in this experiment are Roundup® and Garlon®4 which have the active ingredients Glyphosate and Triclopyr, which are both on the list.

Table 2.1. Herbicides Used In Vegetative Control. "Herbicides Used In Vegetative Control." *USDA Forest Service*. Web. 23 Aug 2011.

Herbicide
2,4-D
Chlorsulfuron
Clopyralid
Dicamba (Vanquish)
Glyphosate (Accord, Rodeo, Roundup, Roundup Pro)
Hexazinone
Imazapic
Imazapyr (Arsenal, Chopper, Stalker)
Metsulfuron Methyl (Escort)
Oxyfluorfen
Picloram
Sethoxydim (Poast)
Sulfometuron Methyl
Triclopyr (Garlon 3A, Garlon 4)

<u>Roundup®</u>

Roundup® brand herbicides were developed by the Monsanto Company in 1974 (Figure 2.1). According to the company's website, "herbicides are key products used in conservation tillage (or no-till) farming, which leaves the soil undisturbed between cropping seasons – therefore being a major force in reducing soil lost to wind and water erosion."[7] They are used worldwide to help control and manage unwanted vegetation growth.

Figure 2.1. Roundup® herbicide used throughout
the experiment.

Roundup® herbicides are non-selective herbicide mixtures which include glyphosate (active ingredient), water, and a patented surfactant system.[8] Non-selective means that there are no known plants that are naturally resistant to Roundup® and therefore the herbicide kills all vegetation it comes into contact with.

The herbicide Roundup® is applied directly to green, growing parts of plants and is then absorbed and translocated through the plant's tissues.[9] Roundup® works by "inhibiting a biochemical pathway important in the normal functioning of plant. By

[7] "The History of Roundup." *Monsanto*. Monsanto Company, 2011. Web. 17 Jun 2011. <http://www.monsanto.com/weedmanagement/Pages/history-roundup-ready.aspx>.
[8] Ibid.
[9] Ibid.

disrupting the pathway, compounds necessary for the plant's survival cannot be made."[10] It inhibits the activity of the enzyme called EPSP synthase, which catalyzes a step in the production of three amino acids essential for plant growth and life (Tyr, Phe, Trp). Upon exposure the plant wilts and turns yellow and brown as the plant's tissues deteriorate. Because the plant is now incapable of regrowth, it will eventually die.

Glyphosate is the active ingredient in Roundup® herbicides (Figure 2.2). It comes in two forms: acid and salt. It is water soluble, odorless, and non-volatile. Depending on the amount used and the other chemicals it is combined with, it can be formulated in low to high toxicity grades.[11]

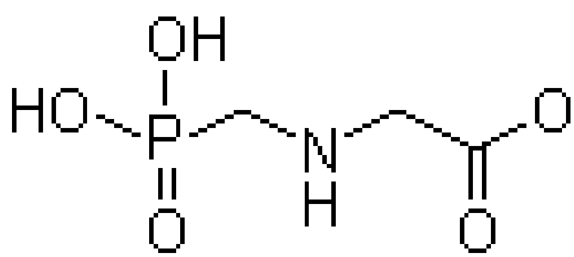

Figure 2.2. Molecular Structure of Glyphosate. "Glyphosate." *LookChem: Glyphosate*. Web. 23 Aug 2011.

In the environment, glyphosate has low to moderate persistence in soil, which it does bond strongly with[12]. This means that it can remain in soil for long periods of time, contaminate the soil and potentially killing any other vegetation within the area. It has low ground water contamination potential, which means that it remains mostly at the ground surface, usually not penetrating deep enough to contaminate water supplies. However, because it remains near the surface, rain and water runoff into bodies of water

[10] "Glyphosate (General Fact Sheet)." *National Pesticide Telecommunications Network*. NPTN, Nov 2000. Web. 4 Jun 2010. <http://www.glifocidio.org/docs/impactos%20generales/ig7.pdf>.
[11] Ibid.
[12] Monsanto Company . *Safety Data Sheet Commercial Product Roundup Ultra® Herbicide*. 2011. Print.

can cause contamination. Glyphosate can be broken down over time by sunlight and water.

<u>Garlon®4</u>

Garlon®4 was developed by the Dow AgroSciences Company (Figure 2.3) in 2006 for the "control of undesirable woody plants and annual and perennial broadleaved weeds on pastures and rangelands, and in non-crop areas such as rights-or-way, military bases and industrial sites."[13] It is a selective herbicide. Selective herbicides "have the ability to kill certain plants without harming others…Resistant plants can survive by metabolizing the herbicides or not absorbing it."[14] Garlon®4 is orange in color and can "effectively control more than 55 woody plants and more than 25 tough-to-control annual and perennial broadleaf weeds in noncrop areas."[15]

Figure 2.3. Garlon®4 herbicide used in the experiment.
"Garlon4." *Gempler's*. Web. 23 Aug 2011.

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[13] "Garlon 4 Herbicide." *Material Safety Data Sheet*. Dow AgroSciences Canada Inc., 08 Apr 2008. Web. 17 Jun 2011. <http://msdssearch.dow.com/PublishedLiteratureDAS/dh_010c/0901b8038010cc6c.pdf?filepath=ca/pdfs/noreg/010-20732.pdf&fromPage=GetDoc>.

[14] "Triclopyr: General Fact Sheet." *National Pesticide Information Center*. NPIC, n.d. Web. 17 Jun 2011. <http://npic.orst.edu/factsheets/triclogen.pdf>.

[15] "Garlon 4 Ultra Specialty Herbicide." *Garlon 4 Ultra Specialty Herbicide*. Dow AgroSciences, n.d. Web. 17 Jun 2011. <http://msdssearch.dow.com/PublishedLiteratureDAS/dh_0061/0901b80380061e1d.pdf?filepath=ivm/pdfs/noreg/010-50595.pdf&fromPage=GetDoc>.

The herbicide Garlon®4 is applied through basal bark, cut-stump, or foliar applications. Basal bark applications "are hand-sprayed to the lower 15 inches of the bark at the base of the tree or brush" and "cut-stump applications are made after tree removal application to the cut stump surface and remaining bark to prevent tree resprouting."[16] Finally, foliar application "allows herbicide application to plant foliage using ground or aerial (helicopter) equipment."[17]

Garlon®4 contains Triclopyr (active ingredient), Kerosene, and propriety surfactants (Figure 2.4). "Triclopyr is used for the control of undesirable woody and herbaceous weeds."[18] Garlon®4's ingredients act as a "synthetic auxin, giving a plant an auxin overdose 1000 times natural levels, which disrupts the hormonal balance and interferes with growth,"[19] starting at a the cellular level first and then spreading throughout the plant until death occurs.

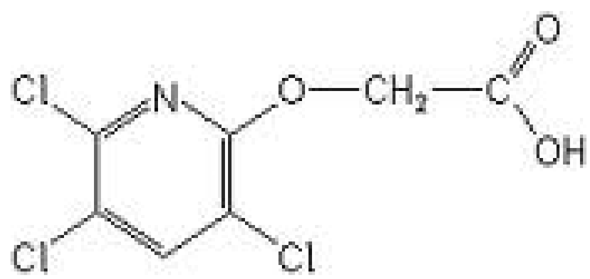

Figure 2.4. Molecular Structure of Triclopyr. "Structure."
Triclopyr Data Sheet. Web. 23 Aug 2011.

In the environment, triclopyr breaks down into several other compounds including carbon dioxide (CO_2). It can also move through the soil and potentially contaminate

[16] "Garlon 4 Ultra Specialty Herbicide."
[17] Ibid.
[18] "Triclopyr: General Fact Sheet."
[19] "Triclopyr." *Environmental Fate of Triclopyr*. Department of Pesticide Regulation, 02 Jan 1997. Web. 17 Jun 2011. <http://www.cdpr.ca.gov/docs/emon/pubs/fatememo/triclopyr.pdf>.

groundwater, however, the half-life in soil ranges from 1.1 to 90 days.[20] Triclopyr is mainly broken down by exposure to sunlight.

Stone and Masonry Materials

Stone and masonry material has been used for centuries to build structures such as buildings, temples, memorials, and houses. These materials were, and in some cases are, still expensive to use. They were originally used by the wealthy to build expansive structures that were meant to last and be around for centuries for future generations to see, live in, and enjoy. Today, brick, limestone, granite, and concrete are all sturdy materials that are used worldwide to build structures of all shapes and sizes.

The main raw material in bricks is ordinarily clay or shale, or a mixture of the two with sand. There is “no rigid scientific definition of brick clays [that] can be formulated, as almost all clays can (technically) be made into bricks.”[21] Clays are classified by their physical properties and how and where they are formed. The two main types of clay are primary and secondary. Primary clays are formed “when parent rocks are altered deep within the earth’s crust by the action of hot gases and water.”[22] This hydrothermal process mixes the clays with fragments of unaltered parent rock, and cause the clays to be generally deficient in plasticity, which makes them difficult to use in brickmaking.[23] Secondary clays are generally used for brickmaking and can be divided into four major subgroups: “river-deposited or alluvial clays (fluiatile), which are formed either by floods or by the settling out of sediments, as in the bend of a river where the speed of the water

[20] "Triclopyr: General Fact Sheet."
[21] Searle, Alfred Broadhead. *Cement, Concrete and Brick.* New York: D. Van Nostrand Co., 1914. 286. Print.
[22] Gurcke, Karl. *Bricks and Brickmaking: A Handbook for Historical Archaeology.* Moscow, Idaho: The University of Idaho Press, 1987. 3. Print.
[23] Ibid.

is reduced; glacial clays, which are produced by the grinding action of glaciers; lacustrine and marine deposits, which are formed by the smaller particles of clay settling out, in lakes or the sea; and windborne deposits."[24] Clay minerals are the crystalline particles that make up clay, and are essentially hydrous aluminum silicates in the form of "flat or warped plates, tubes, or chains separated by water, which acts as a lubricant between the crystals"[25] and adds to the plasticity of the material. The plasticity in the material is what causes the clay to shrink during the drying process, however, if not done properly, the shrinkage could be too extreme rendering the brick useless or if not enough free-water is removed from the material, the brick would still be in a paste form. [26] In the United States there are five basic steps in making a brick: (1) mining- frequently called "winning," (2) preparation, (3) molding- sometimes referred to as "forming," (4) drying, and (5) firing- frequently referred to as "burning."[27] They are fired in a kiln and "the shrinkage on drying which clay pastes undergo is due to the removal of the water surrounding (and possibly penetrating) each particle, with the result that, as this water evaporates the particles are brought nearer together, until finally they are as close as their shape permits."[28] There can also be impurities in the clay that can affect the outcome of the brick and the hardness, sturdiness, color, etc. of the material. There are many types of clays, and because of the different varieties there can be many different possibilities for brick formation.

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[24] Ibid.
[25] Ibid, 4.
[26] Searle, Alfred Broadhead. 300.
[27] Gurcke, Karl. 4.
[28] Searle, Alfred Broadhead. 301.

Limestone is a sedimentary rock that occurs naturally throughout the world and "makes up approximately 10 percent of the total volume of all sedimentary rocks."[29] It is composed of the mineral calcite (calcium carbonate) whose primary source is marine organisms. "These organisms secrete shells that settle out of the water column and are deposited on ocean floors as pelagic ooze. Secondary calcite may also be deposited by supersaturated meteoric waters (groundwater that precipitates the material in caves)."[30] Limestone is found in many forms and is classified by its origin, chemical composition, structure, and geological formation. Pure limestone is white or almost white in color. However, "because of impurities, such as clay, sand, organic remains, iron oxide and other materials, many limestones exhibit different colors, especially on weathered surfaces" and they "may be crystalline, clastic, granular, or dense, depending on the method of formation."[31] Limestone is quarried and is used extensively as an aggregate in building and construction because it is "readily available and relatively easy to cut into blocks or more elaborate carving. It is also long-lasting and stands up well to exposure. However, it is a very heavy material."[32] It is also expensive. "It has been used as an aggregate in lime-based concrete since Roman times, and, more recently, in cement-based concrete."[33] Today, it is used for roadbeds, landscape and building construction, and cement manufacturing. Despite its durability, limestone is "vulnerable to acids,

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[29] "What is Limestone>." *GraniteLand.* GraniteLand.com. n.d. Web. 6 Jul 2011. <http://www.graniteland.com/infos/home/limestone>.
[30] Ibid.
[31] Ibid.
[32] Ibid.
[33] Oates, Joseph A.H. *Lime and Limestone: Chemistry and Technology, Production and Uses.* New York: Wiley-VCH, 1998. 1. Print.

making acid rain a problem when it occurs in places where limestone is used extensively. The acids in the water can wear away the details of statues and other art."[34]

Concrete building blocks are used in construction of exterior walls for building. They are inexpensive, and some of the advantages of concrete blocks are: "(1) their hollow form results in a saving of materials over brick or stone masonry, this often amounting to from 20 to 50 percent, (2) the cost of laying concrete blocks is less than for brickwork, this is due to the fact that the blocks, being larger, have a much smaller number of joints, and require less mortar, and, being hollow, are of less weight than solid brick work, and (3) a wall, properly constructed of good concrete blocks, is as strong or stronger than a brick wall of equal thickness."[35] The concrete mix for cement blocks "shall not be richer than one part of cement to six parts of volume of combined aggregate."[36] The size of the coarse aggregate generally used is 6 to 12 mm and "sixty percent fine and forty percent coarse aggregates" is recommended.[37] Portland cement is the most commonly used cement in this process. Cement is "a material which binds together solid bodies (aggregate) by hardening from a plastic state."[38] Portland cement includes the components of tri- and di-calcium silicates, and is commonly referred to as simply 'cement'.

There are many types of granite, but in general the term granite refers to "quartz-bearing (>60 wt% SiO_2) plutonic igneous rocks."[39] Granite is "a common and widely-

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[34] "What is Limestone."

[35] Reid, Homer Austin. *Concrete and Reinforced Concrete Construction*. New York: The Myron C. Clark Publishing Co., 1907. 855. Print.

[36] Varghese, P.C. *Building Materials*. New Delhi: Prentice-Hall of India, 2005. 26. Print.

[37] Ibid.

[38] Bye, G.C. *Portland Cement: Composition, Production and Properties.* 2nd. London: Thomas Telford Publishing, 1999. 1. Print.

[39] Chen, Guo-Neng, and R.H. Grapes. *Granite Genesis: In-Situ Melting and Crustal Evolution*. Dordrecht: Springer, 2007. 4. Print.

occurring group of intrusive felsic igneous rocks that form at great depths and pressures under continents."[40] It consists of orthoclase and plagioclase feldspars, quartz, hornblende, biotite, muscovite and minor minerals such as magnetite, garnet, zircon, and apatite, and the average density of 2.75 g/cm^3 with a range of 1.74 to 2.80.[41] There is much debate over the origin of granite: magmatic theory, which states that granite is derived by the crystal fractioning of magma, and the granitization theory, which states that granite is formed in place by extreme metamorphism.[42] Physical properties of granite include color, weight, strength, hardness, and porosity. Color "depends chiefly on the relative abundance of the dark ferromagnesian mineral or minerals (biotite or hornblende, or both) and the character of the feldspars. Those granites containing a large proportion of biotite or hornblende are of darker gray color than those containing but little."[43] The specific gravity of granite ranges from 2.593 to 2.731, and the ultimate compressive strength of granite is from 18,000 to 34,000 pounds per square inch. "Granite usually contains about 0.8 percent of water and is capable of absorbing about 0.2 percent more."[44]

Salt and Efflorescence

Efflorescence is defined by the National Park Service as "the outward migration and precipitation of salts on the surface from within a porous material."[45] It is the result of capillary action pulling soluble salts up from the ground into the masonry material. It

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[40] "What is Granite." *GraniteLand.* GraniteLand.com. n.d. Web. 3 Aug 2011. <http://www.graniteland.com/infos/home/granite>.
[41] Ibid.
[42] Chen, Guo-Neng, and R.H. Grapes. 3.
[43] Watson, Thomas Leonard. "Granite of the Southeastern Atlantic States." *Department of the Interior United States Geological Survey Bulletin 426*. (1910): 20. Print.
[44] Watson, Thomas Leonard. 21.
[45] "Archeology Program: Managing Archeological Collections." *Archeological Program*. National Park Service, 10 Feb 2009. Web. 3 Aug 2011. <http://www.nps.giv/history/archeology/collections/glossary.htm>

usually appears as a white haze on the exterior of the surface.[46] It is not hazardous. As water evaporates from the surfaces, mineral deposits are left, which may cause the formation of efflorescence. "Some efflorescence is temporary, and will be removed by rain. Other types may disappear for a while, but return periodically, and some require considerable and repeated efforts to eliminate."[47] Efflorescence is generally a visual problem, however, if crystals form within the material spalling can occur, which can lead to further deterioration problems.

[46] Grimmer, Anne E. *Keeping It Clean: Removing Exterior Dirt, Paint, Stains and Graffiti from historic Masonry Buildings.* U.S. Department of the Interior, 1988. 5. Print.
[47] Ibid.

CHAPTER 3

RESEARCH

It was important to develop a strategy for designing an experiment that would accurately reflect the methods most often used to apply herbicide, the most commonly used herbicides, and the types of masonry and stone material that the herbicides were coming into contact with. It was also important to determine what effects on the masonry and stone material were the most important to focus on, as the anticipated effects would help decide the tests to be run and the equipment to be utilized.

The Big Question

The main questions being examined in this study are: 1) does the contact of herbicides on stone and masonry cause harmful effects to the stone and masonry structure, and, if so, 2) do the herbicides cause increased rates of deterioration in the stone and masonry materials that could result in structural failure? The focus of this study is on masonry and stone materials and how they react when they come into contact with herbicides. This question is being asked at historic sites by the U.S. National Park Service and at other locations where herbicides are used to control unwanted vegetation near historic structures. The people responsible for preserving the historic structures and maintaining the landscapes want to know if the herbicides they use are accelerating the deterioration to the materials they are trying to preserve and protect.

Previous Studies

Over the past twenty-two years there have been only two studies that examine the effects of herbicides on masonry material. In 1989, Linda Anne Cook wrote her Master's thesis on "The Effects of Herbicide on Masonry: Products, Choices & Testing" at Columbia University, and, in 1999, Catherine Camille Dewey presented her Master's thesis, "An Investigation into the Effects of an Herbicide on Historic Masonry Materials" at the University of Pennsylvania. Both studies used the available technology at the time to examine how herbicides affect the structure of masonry materials and demonstrated that herbicides caused negative effects to the masonry. The work by Cook and Dewey provide a baseline for this thesis experiment by: 1) showing that there are negative effects that occur when masonry materials are contacted by herbicides and 2) it providing a foundation from which to develop a new experimental design that builds upon their lessons and suggestions.

Cook received her Master of Science in Historic Preservation at the Graduate School of Architecture, Planning and Preservation at Columbia University. Her thesis was one of the first major studies conducted to determine the effects herbicide had on stone and masonry materials. Cook states that "Choosing the most effective herbicide may not be the best choice for the masonry. Finding suitable herbicides that meet the determined criteria for both removing unwanted vegetation and having no detrimental effect on the masonry is derived from assessing laboratory testing."[48] Her goal was "to ascertain whether commercial herbicides are mechanically and chemically destructive to masonry."[49] Cook chose three herbicides to test on brick, limestone, and mortar: 1) a

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[48] Cook, pg. 2.
[49] Cook, pg. 63.

saturated solution of sodium tetraborate (at room temperature), 2) Weed-B-Gon® herbicide (at 0.6% solution, the manufacturer's suggested solution strength and 0.3% solution; and 3) Roundup® herbicide (at 5.5% solution , the manufacturer's suggested solution strength, and –2.7% solution), with distilled water as a control.[50] The brick, limestone, and mortar samples were completely submerged in the herbicide solutions for 14-16 hours, drained, dried in preheated ovens for 4-5 hours, allowed to cool to room temperature, and then the cycle was repeated in new sample solutions for a total of 10 cycles. Samples were weighed every 2 cycles and the pH of the sample taken every 3 cycles. Cook was specifically looking for salt crystallization, pH change in the material, and discoloration. Cook found that all the samples experienced a weight change, and "in most cases the weight decreased," which could be attributed to the mechanical deterioration of the masonry material in the herbicide.[51] She observed that minor surface pitting and imbedded salt crystals, especially in the Roundup® solutions, and discoloration was more discernable in the masonry exposed to the Weed-B-Gon® solutions. Cook's experiment showed that herbicides affected the stone, and "the stronger the strength of the solution, the greater the quantity of efflorescence"[52].

In 1999, Catherine Camille Dewey received her Master of Science degree in Historic Preservation at the University of Pennsylvania. Her thesis was the next step in the study of herbicide effects on masonry materials. "The aim of this testing program was to evaluate the effects of the herbicide Round-up® and its surfactants have on selected masonry materials (brick, limestone, and sandstone) from historic structures and

[50] Cook, pg. 64.
[51] Cook, pg. 75.
[52] Cook, pg. 76.

archaeological sites."[53] Two Roundup® solutions, a surfactant solution, and a control were used in this experiment: 1) Roundup® herbicide mixed with tap water – 17% solution; 2) Roundup® mixed with tap water – 34% solution; 3) tap water and ethoxylated tallow amine at 2%; and 4) tap water as the control. [54] The brick, limestone, and sandstone samples were immersed completely for 24 hours and dried for another 24 hours. "(S)even cycles were run on the weathered stone [sandstone]and brick and ten cycles were run on the limestone and new brick to ensure results."[55] Dewey was specifically looking for changes in the samples' mineralogy, porosity, weight, color, pH, and the formation of salt. Her experiments yielded the following observations: increased pore space, discoloration, pH and weight change, pitting/surface loss, and the materials became softer and more friable. Dewey concluded that, "The damage to masonry caused by glyphosate is three-fold. It first attacks calcareous stone by acid dissolution. Secondly, it and its solvent, in this case water, introduce or redeposit soluble salts. Thirdly, in the presence of calcium – a major component of building stones – it forms insoluble salts."[56] And due to the damaging effects witnessed in her experiment, Dewey suggests that "before the plants have reached maturity, mechanical removal is the best option."[57]

Cook and Dewey's studies helped form the basis for this study. They brought focus to an important issue: the negative effects of herbicides on historic sites. This research advances their experiments by using newer technology to better quantify the observations and exposures more likely to occur in real situations.

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[53] Dewey, pg. 47.
[54] Dewey, pg. 51-52.
[55] Dewey, pg. 51.
[56] Dewey, pg. 84.
[57] Dewey, pg. 87.

National Park Service Sites Survey

A survey was created, entitled 'The Use of Herbicides Near Masonry,' (Appendix A) and distributed to 470 National Park Service (NPS) facility managers by the NPS Historic Preservation Training Center (HPTC), on June 21, 2010. The survey asked if herbicides were used to remove unwanted vegetation at their site. If participants answered yes, they were directed to a series of questions about the type of herbicide used, at what concentration is it applied, how it is applied, and what type of historic feature it is used on or near. This survey could be completed for up to three historic features at a site. If participants answered no, they were asked what method is used at the site to remove unwanted vegetation (examples included mechanical removal, hand removal, and no treatment). The goal of the survey was to collect data in order to design an experiment based on the most widely used herbicides and the most common historic masonry materials found at National Park sites. The survey closed on July 1, 2010.

In the ten days that the survey was available, NCPTT received 98 survey responses from National Park Service sites across the United States; a 21 percent return. In total, 36 states were represented by at least one NPS site, including two from Hawaii - Kalaupapa National Historical Park and Kaloko Honokohau National Historical Park; and one from Alaska - Klondike Gold Rush National Historical Park. In addition, the National Capital Parks-East in the District of Columbia also provided feedback, as did three NPS sites located in U.S. territories: the War in the Pacific National Historical Park in Guam, the Christiansted National Historic Site in the Virgin Islands, and the American Memorial Park in Saipan, Marianas Protectorate. Ten sites opted out of providing their site name/location.

Results from the survey revealed that 79 percent of the sites that responded used herbicides as a method of removing unwanted vegetation. Of the sites that do not use herbicides, 54 percent remove unwanted vegetation by hand, 27 percent use a form of mechanical removal, and 19 percent do not treat the site but rather leave the vegetation in place.

The survey found that 58 percent of the NPS site responses use Roundup®, 22 percent use Garlon®4 and the remaining 20 percent use other varieties of herbicides such as Accord® and Surge® (Figure 3.1).

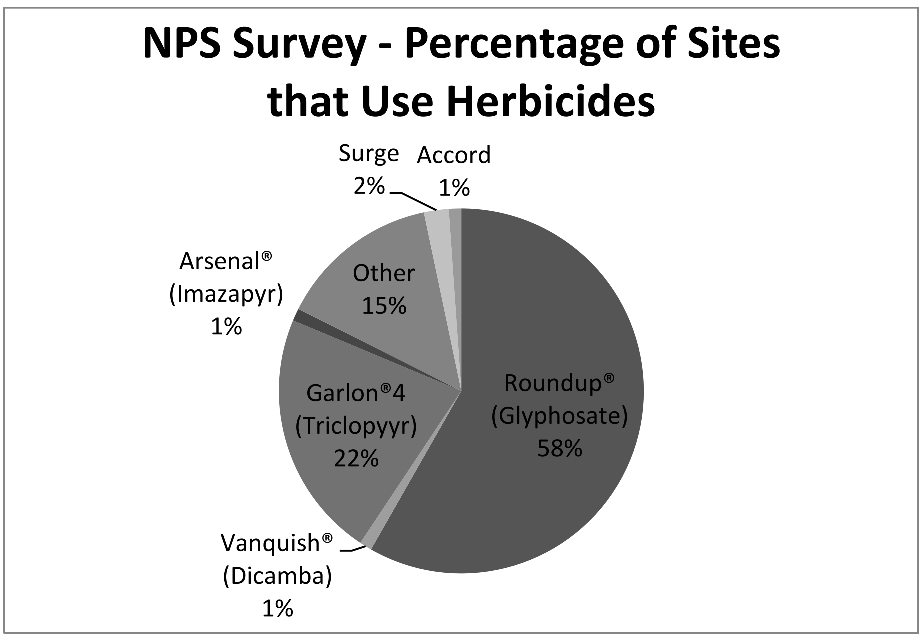


Figure 3.1. NPS Survey Results for the Types of Herbicides Used at NPS Sites.

Participants were also asked how the herbicide is applied to the foliage and how often it is applied. Seventy-two percent of herbicide users spray it onto the foliage, 26 percent use the spot treatment on cut surfaces, and 2 percent hand scrub the herbicide solution onto the surface. Meanwhile, 37 percent of NPS sites apply the herbicide as needed, 18 percent apply it twice a year, 16 percent apply it annually, 8 percent apply it monthly, 3 percent apply it daily and the remaining 18 percent apply it at a variety of other times throughout the year. It was also found that 88 percent of herbicide users apply the manufacturer's recommended concentration to unwanted vegetation whereas 12 percent mix their own concentration levels of solution.

For the purpose of this survey, a historic feature is a manmade, built structure that is at least 50 years old and has historical significance. This includes, but is not limited to, houses (29 percent of respondents had this feature at their site), outbuildings (6 percent), forts (11 percent), monuments (13 percent), cemeteries (9 percent), ruins (4 percent), paths/trails (3 percent), walls (6 percent), historic districts (1 percent), other buildings such as churches (8 percent), and miscellaneous features (8 percent). Participants were required to specify the type of historic stone and masonry materials that are present at their site. The choices that were provided were brick, concrete, sandstone, limestone, marble, and granite, with a choice of "other" if their material was not listed. The results are as follows: 24 percent brick, 14 percent limestone, 13 percent granite, 11 percent concrete, and 5 percent marble. Other material results (33 percent of responses) included features that did not fall directly under the category of stone or masonry, but may be useful for future experiments. These included coquina, rock/soil, wood, tabby, adobe, coral, bronze, stucco, and steel (Figure 3.2).

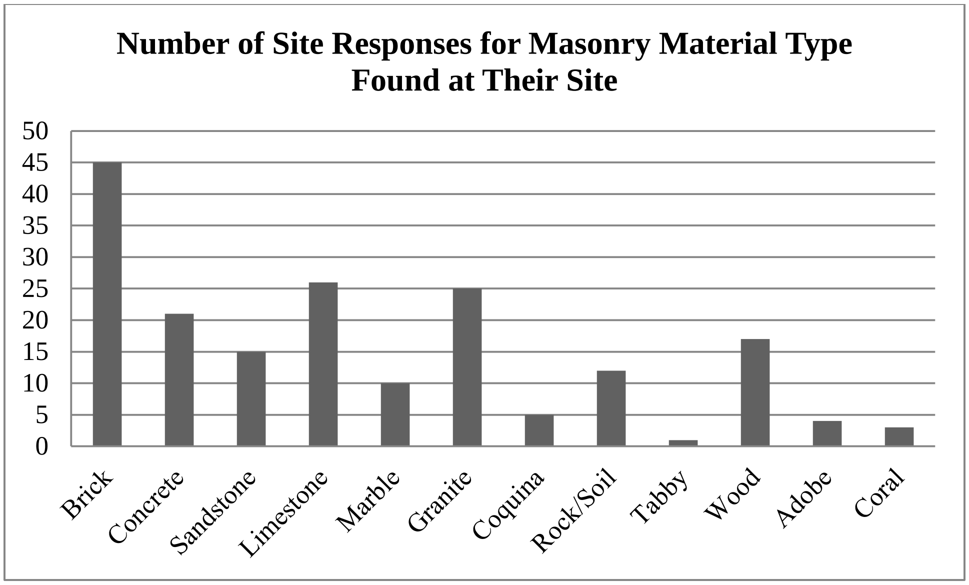


Figure 3.2. Masonry Type Responses. The number of site responses for masonry material type found at each NPS site that responded.

Integrated Pest Management (IPM) is defined by the National Park Service as "an approach to pest management that employs physical, mechanical, cultural, biological and educational tactics to keep pest numbers low enough to prevent intolerable damage or annoyance. In an IPM program, the least toxic, effective management options are utilized."[58] So, in addition to questions about the use of herbicides, survey participants were asked if an IPM system was in place at their respective site for controlling unwanted vegetation growth. Sixty-nine percent of the sites replied that there was an IPM system in place at the site, 16 percent said there was not, and 14 percent chose not the respond to this question.

Preparation

Based on the results from the survey, experiments were designed to study the effects of Roundup® and Garlon®4 herbicides on historic brick, limestone, granite, and

[58] "Integrated Pest Management: Frequently Asked Questions." National Park Service. 15 July 2010. <http://www.nps.gov/nero/ipm/ipmfaq.htm>.

concrete. These were the most commonly used herbicides and the most common masonry and stone materials located in National Park sites.

Roundup® herbicide was purchased at the local hardware store. Garlon®4 was purchased directly from the manufacturer.

The historic brick was gathered from the ruins of a historic house in Cloutierville, Louisiana. Because the house had burned down, special care was taken to ensure that the bricks that were collected were not glazed or burned from the heat of the flames. NCPTT had previously cut samples of Indiana limestone for experimental use, leftover from a previous study. A concrete block was the source of cement for this project. The granite is from Elberton, Georgia.

Samples of each material were cut using a coring machine with a diamond tip and a saw blade (Figure 3.3).

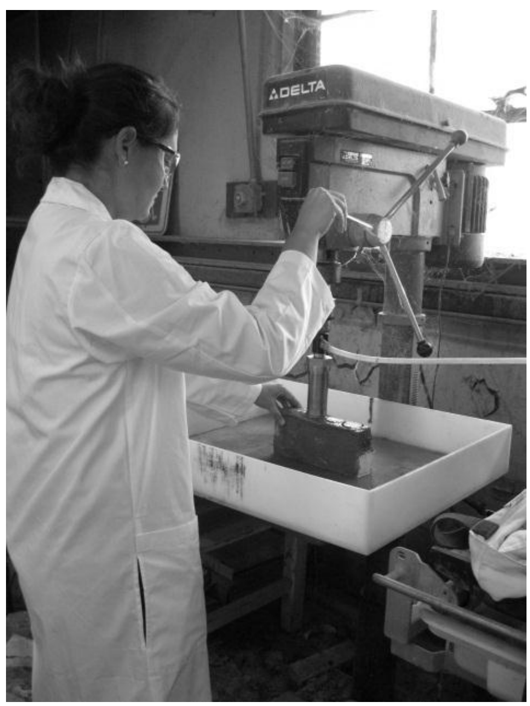


Figure 3.3. Coring Out Brick Samples.

Concrete samples were cored and then carefully chiseled out of the concrete block (Figure 3.4 and 3.5) Each sample is 3.81 centimeters (cm) in diameter. The approximate thickness of each of the samples differed. Because herbicides come into contact with the surface of the materials, each sample was cut with a face that was on what would be the exposed side of the stone or masonry material.

Figure 3.4. Chiseling Out Concrete Samples After Coring.

Figure 3.5. Trying Not to Damage the Surface of the Concrete Samples.

Approximately 15 samples were cut of each material.

All the samples were then polished using a grinder to smooth the edges and bottom face so that each would lay flat and even (Figure 3.6). The exposed surface was not polished.

Figure 3.6. Polishing and Evening Out the Bottom Face of a Brick Sample.

Each sample then received an identification number written on the back with a Sharpie® (preliminary test samples) or ‘engraved’ into the back face. Each sample was then washed using deionized water to remove any dust and grime from the surface and placed in an oven at 70 degrees Celsius for 2 hours to dry. After this process, gloves were used at all times when handling the samples so as not to contaminate them with dirt or oil from skin and hands.

CHAPTER 4

PRELIMINARY EXPERIMENT

A preliminary experiment was conducted to determine if physical changes would be observed when stone and masonry materials came into contact with herbicides over an exposure of several days. For the main experiment to be meaningful, there needed to be some observable change to the stone and masonry materials. Also, the data generated from this preliminary experiment provided useful information for developing the design of the main research.

Safety

Safety was a high priority in the design and implementation of both the preliminary experiment and main research. To reduce manual exposure to toxic materials in the laboratory and reduce the opportunities for sample contamination, all people handling the herbicides and masonry samples wore gloves at all times. Eye protection and laboratory coats were also worn when handling the herbicide solutions. Roundup® and Garlon®4 were poured under a fume hood to reduce the risks of inhalation.

Preliminary Experiment - Design

This preliminary experiment used brick, concrete, and limestone, and Roundup® herbicide, with deionized water as the control. Four samples of each material were used and labeled as follows, using a Sharpie®:

NBa = water, brick, sample 1
NLa = water, limestone, sample 1
NCa = water, concrete, sample 1
RBa = Roundup®, brick, sample a

RBb = Roundup®, brick, sample b
RBc = Roundup®, brick, sample c
RLa = Roundup®, limestone, sample a
RLb = Roundup®, limestone, sample b
RLc = Roundup®, limestone, sample c
RCa = Roundup®, concrete, sample a
RCb = Roundup®, concrete, sample b
RCc = Roundup®, concrete, sample c

The first letter in the identification system represented the type of herbicide each sample would be exposed to ("R" means that Roundup® herbicide would be used and 'N' means that "No" herbicide would be used, instead water would be used as a control). The second letter represented the type of stone or masonry material (brick, limestone, concrete), and the third letter represents the replicate (a, b, c) as three samples of each combination were tested.

Nine petri-dishes were filled halfway with Roundup® herbicide and 3 petri-dishes were filled halfway with deionized water. Each masonry sample was placed face-down in the appropriate solution. Placing the samples in the exposure solutions in this way mimicked the outer surface exposure to the herbicide. The masonry samples were then left in the solution for 24 hours. Each Petri-dish was refilled, as needed, with its appropriate solution to maintain a constant exposure level. After 24 hours, the samples were removed from the solutions and left to air dry for 24 hours, thus completing one cycle. All samples completed 5 cycles.

For visual reference, photographs of the samples were taken before immersion in the exposure solutions and immediately following completion of all five cycles (Figure 4.1). Photographs were taken of each sample using a Nikon D5000 camera with a Nikon DX 35 mm lens. The camera was positioned 12.75 inches above the surface of the table where the samples were placed. Eiko Supreme Photoflood lights, at ECT 120 volts, were positioned in the full upright position above the camera in order to ensure full lighting and no shadows on the surface of the samples.

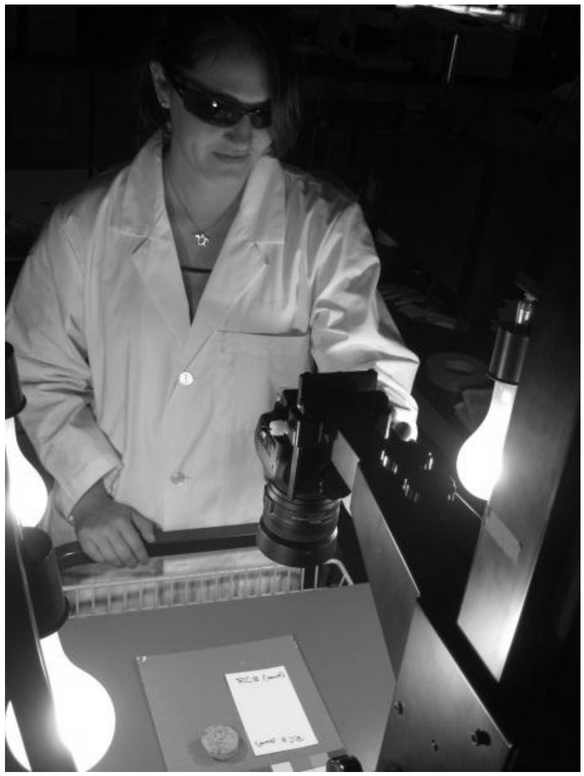
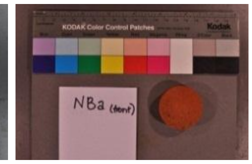
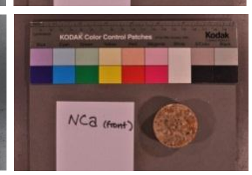
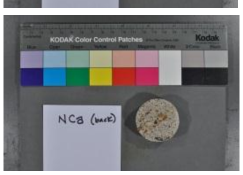
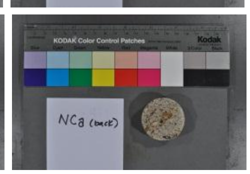
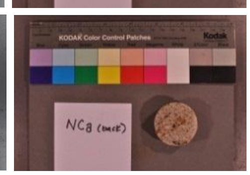
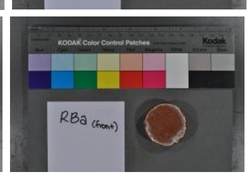
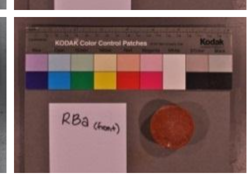
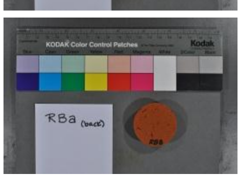
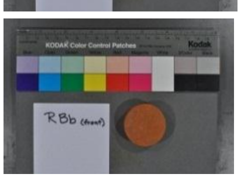
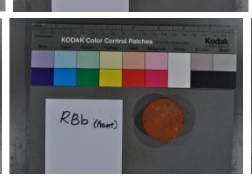
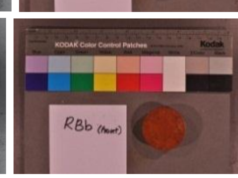


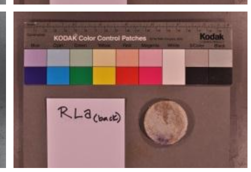
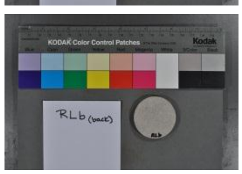
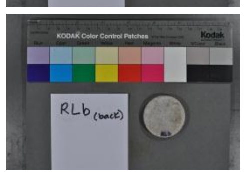
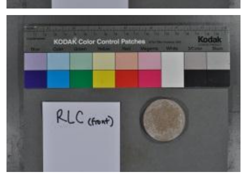
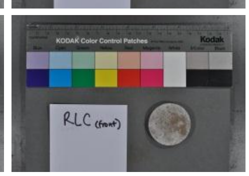
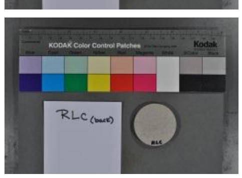
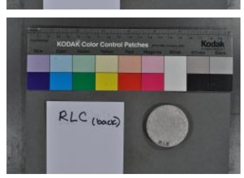
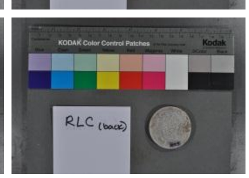
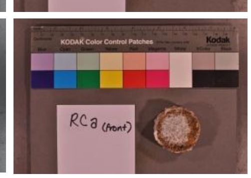
Figure 4.1. Photographing Samples Before Treatment in the Laboratory.

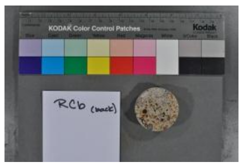
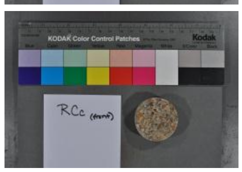
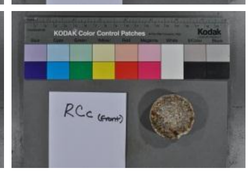
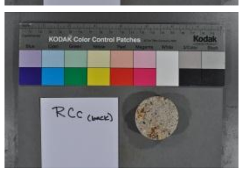
Data Results

After the first cycle, efflorescence had formed on all concrete and limestone samples, as well as on two of the three brick samples (RBa and RBb). No efflorescence formed on the control samples. A glossy sheen was observed on the fronts of the limestone samples that were in the Roundup® solution. All samples exposed to herbicide were observed to have developed efflorescence by completion of the second cycle.

The following photographs were taken of the samples at different times throughout the preliminary experiment to document any physical change the material undergo upon exposure to the herbicide.

Sample ID	**0 Hours**	**48 Hours**	**144 Hours**	**204 Hours**
NBa (front)				
NBa (back)				
NLa (front)				
NLa (back)				
NCa (front)				
NCa (back)				
RBa (front)				
RBa (back)				
RBb (front)				

Sample ID	0 Hours	48 Hours	144 Hours	204 Hours
RBb (back)				
RBc (front)				
RBc (back)				
RLa (front)				
RLa (back)				
RLb (front)				
RLb (back)				
RLc (front)				
RLc (back)				
RCa (front)				

Sample ID	**0 Hours**	**48 Hours**	**144 Hours**	**204 Hours**
RCa (back)				
RCb (front)				
RCb (back)				
RCc (front)				
RCc (back)				

Analysis and Conclusion

This preliminary experiment reinforced information from previous studies that physical changes occur when stone and masonry materials come into contact with the herbicide, Roundup®. The exposure to Roundup® caused copious efflorescence to develop on the surfaces of all the herbicide-exposed samples (Figure 4.2).

Figure 4.2. Before and After Samples of Concrete. The sample on the left was not exposed to any herbicide and the sample on the right was one of the samples used in the preliminary experiment.

Roundup® herbicide affected brick, concrete, and limestone when they are exposed for approximately 48 hours and allowed to go through two wet/dry cycles. However, this scenario is not realistic. In no known cases are historic features sitting for days at a time in herbicide. But, this test demonstrated the extreme changes that can be observed in 10 days.

CHAPTER 5

METHODS

Upon conclusion of the preliminary experiment, an experiment was designed that more comprehensively demonstrated how herbicide affects stone and masonry materials under an exposure scenario that is closer to real circumstances. Spraying herbicide on or around masonry is an action that is normally performed only a few of times a year. The historic sites that are exposed to herbicides are outdoors and affected by sunlight and weather. This experiment was designed to observe the effects of herbicides on masonry over an extended period of time and with simulated weather (moisture, UV light).

QUV Accelerated Weathering Tester

To simulate exposure to weather over an extended period of time, NCPTT's QUV Accelerated Weathering Tester (Q-panel laboratory ultraviolet tester or QUV) was implemented. A QUV is an accelerated weathering tester that reproduces the effects of weather conditions that occur over months or years outdoors within just a few days.[59] "Its short wavelength ultraviolet light and moisture cycles realistically simulate the damaging effects of sunlight, dew, and rain."[60] Using the QUV enables tests to simulate, long-term outdoor conditions in shorter periods of time. The QUV Accelerated Weathering Tester – Model QUV/Spray Q-Panel developed by Lab Products was used throughout this experiment (Figure 5.1).

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[59] "QUV Weathering Tester." Q-Lab Corporation, 2008. Web. 7 Jun 2011. <http://www.q-lab.com/QUV.html>.

[60] Ibid.

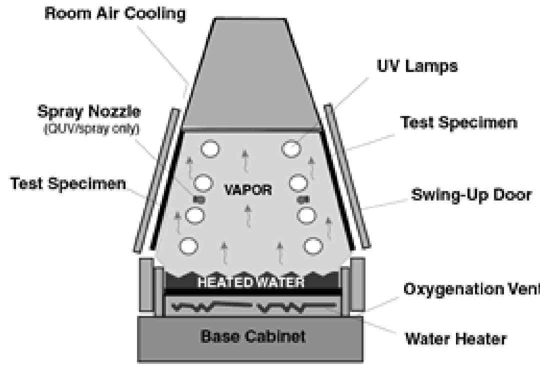

Figure 5.1. QUV Moisture Simulation. "QUV Moisture Simulation."
QUV Moisture Simulation. Web. 23 Aug 2011.

A QUV uses fluorescent ultraviolet (UV) lamps to simulate damaging sunlight. “Although UV light makes up only about 5% of sunlight, it is responsible for most of the sunlight damage to polymer materials exposed outdoors,” and therefore, “it is only necessary to reproduce the short wavelength UV for testing polymer degradation.”[61]

The QUV can be programed to produce cycles of wetness alternating with cycles of UV, much like natural weathering cycles. “Studies have shown that condensation in the form of dew is responsible for most outdoor wetness,” and “is more damaging than rain because it remains on the material for a long time, allowing significant moisture absorption.”[62] A spray system can simulate rain. The temperature inside the weathering tester can be controlled and maintained throughout the experiment.

Using the QUV, the masonry samples were exposed to temperature conditions, moisture, and ultraviolet light that simulated an outdoor environment. This experimental exposure environment was controlled and monitored.

[61] "QUV Weathering Testers."
[62] Ibid.

Experiment Procedure

For this study, historic brick, Indiana limestone, granite from Elberton, Georgia, and concrete samples were used. All samples were cores with a 3.81 centimeter (cm) diameter. The average thickness of the samples is as follows: brick 1.27 cm, limestone 0.635 cm, concrete 0.794 cm, and granite 1.588 cm. The face of each sample was not altered prior to experimentation. The size of the samples enabled multiple tests to be conducted that were the correct dimensions for the QUV plates during the weathering cycles. The front face of each masonry sample was treated with 2 squirts (approximately 2.50 milliliters or mL) from the bottle of herbicide (Roundup® or Garlon®4) or control (deionized water) every 200 hours, at the end of a condensation cycle while in the QUV. The QUV ran on 8 hour cycles (4 hours light and 4 hours dark). Every 400 hours, the masonry materials were evaluated for chemical and/or physical changes. The samples remained in the QUV for a total of 800 hours.

The solutions used for this experiment were ready-to-use Roundup®, directly from the commercial container, and pure Garlon®4, purchased from the manufacturer. Deionized water was used for the control samples. To ensure a consistent spray, each liquid solution was poured into one of three identical spray bottles. The nozzles were adjusted to the “spray” capability and tested to ensure that they were all squirting the same volume over the same surface area (Table 5.1).

Table 5.1. Average Amount of Spray. The average amount of spray that was applied to the samples in 2 squirts from each spray bottle.

Herbicide	Beaker Mass	Beaker + Herbicide Mass	Solution Mass per 2 squirts
Roundup®	69.94 g	72.45 g	2.51 g
Garlon®4	66.13 g	68.66 g	2.53 g
DI Water	49.25 g	51.53 g	2.28 g

Between treatments, the bottles were stored together in the same location within laboratory and no additional solution was added to the bottles.

The masonry samples were sprayed after they had been randomly placed in the sample holders for the QUV. Colored tape was placed on the back of each aluminum sample holder to distinguish the different treatment types when the samples were removed for spraying. The markings were: green for Roundup®, orange for Garlon®4, and blue for DI water. For spraying, each QUV rack was removed from the QUV and placed face up over a small wash basin so that any excess liquid spray would not run into other samples but rather into the basin for disposal. A 20.32 cm x 25.4 cm plastic sheet was placed over each sample and an additional 8 inch x 10 inch plastic sheet was held at a 90 degree angle to the covered sample to minimize overspray contamination of other objects (Figure 5.2). The sample was then sprayed twice from a 15.24 centimeter distance. Once the sample surface was dry, the holder was placed back into the QUV. This process was repeated for each sample every 200 hours.

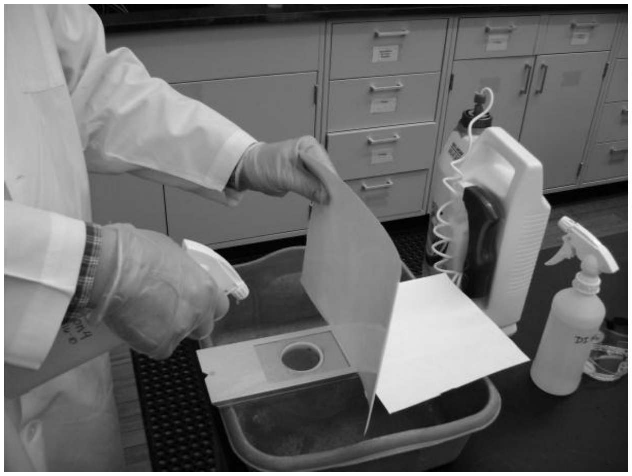

Figure 5.2. Spraying Garlon®4 herbicide onto a Sample Before Placing It into the QUV for Weathering.

The QUV was programmed to maintain a temperature of 50 degrees Celsius and remain running for 800 hours. The QUV ran on alternating wet/dry cycles (4 hours each).

At 400 hours and 800 hours, the samples were removed from the sample holders. Pins were placed in a foam sheet on trays to elevate each sample and minimize contact with the hard, flat surface that could knock off or destroy any evidence (e.g., efflorescence) of change in the sample. Each sample was photographed for visual documentation and then examined for chemical and physical changes.

<u>What Changes Were Expected</u>

It is important to have knowledge of background information when designing an experiment. This information helps develop expectations of the types and magnitude of changes that will be observed during and at the conclusion of the study. These expectations help determine what equipment needs to be used and what tests should be performed to investigate any changes.

Based on previous studies by Cook (1989) and Dewey (1999), and the preliminary experiment performed by the author, it was known that efflorescence is likely to form, especially when exposed to Roundup® over extended periods of time. Increases in mass are likely to occur as the herbicide is initially absorbed into the pores of samples. However, over time, the herbicides will start to deteriorate the material, causing pitting and loss of material, resulting in an eventual decrease in mass. Surface changes are expected, such as pitting and deterioration. Changes in hardness are expected as the samples are negatively affected by the chemicals in the herbicide and the chemical bonds holding the material together are weakened, resulting in a more brittle structure that is likely to lead to eventual structural failure. Discoloration may also occur from chemical reactions on the surface of the samples. Several methods for observing, testing, and measuring changes were used in this experiment to investigate the reactions of the sample materials to herbicides.

Laboratory Equipment

The samples were evaluated by techniques for determining physical and chemical changes to the material caused primarily by the herbicides. The methods used for quantifying the physical changes in the samples were: laser profilometery, absorption, color, mass, photography, and visual ranking. The methods used for testing the chemical changes in the samples were: ion chromatography and x-ray diffraction.

Photography

Photographs of each of sample were taken before, during, and upon completion of the experiment exposure. A visual change was expected to occur as the herbicide was applied to the masonry materials, and this visual change and any subsequent visible or physical changes to the materials were documented as the experiment progressed.

All samples were photographed using a Nikon D5000 camera with a Nikon DX 35 mm lens. For each photo, the camera was located 12.75 inches above the table surface where the sample was resting. Eiko Supreme Photoflood (ECT 120 volts) lights were used to light the area, and the lights were positioned in exactly the same position in their full upright position while photographs of the samples were being taken.

Mass

The mass of each sample was measured before, during, and at the conclusion of the experiment. An increase in mass of each masonry material was expected due to the presence of efflorescence/salt buildup. By tracking the mass of each sample, it helped determine any decrease or accumulation of mass.

The samples were weighed on a Mettler Toledo Classic Plus (AB204-S/FACT) scale and weighed to the nearest hundredth of a gram. The Mettler Toledo Classic Plus (AB204-S/FACT) has a readability of 0.1milligrams (mg) to 0.01 mg and a repeatability (standard deviation) of 0.1 mg. At each weight measurement, each sample was weighed three times and the mean of those measurements was calculated to be the weight of the sample at that time.

pH – Litmus Paper

The pH of each sample will was tested before, during, and after the experiment. Litmus paper was used for this test. Due to the high acidity in the herbicide and the low acidity in the masonry materials, it was expected that the masonry materials would develop a higher acidity level as herbicide was applied to them and absorbed. This change in acidity might lead to accelerated rates of deterioration of the materials.

pH is the measure of acidity or alkalinity of an aqueous solution, with 7.0 being neutral, a measurement of less than 7.0 being acidic, and a measurement greater than 7.0 being basic/alkaline.

Colorimeter

Colorimetry gives a numerical value to a color in order to be used to quantitatively compare colors. These values can also be used to compare the initial color of a sample to the color of the sample after treatment. Color is a mixture of hue, lightness, and saturation. Hue is the term used in color for the classifications of red, yellow, blue, etc. By mixing these hues numerous colors can be created on a color wheel. Lightness can be measured independently of hue and can be separated into bright and dark colors. Saturation is also separate from both hue and lightness. It is the vividness of a color.[63] (Figures 5.3 and 5.4)

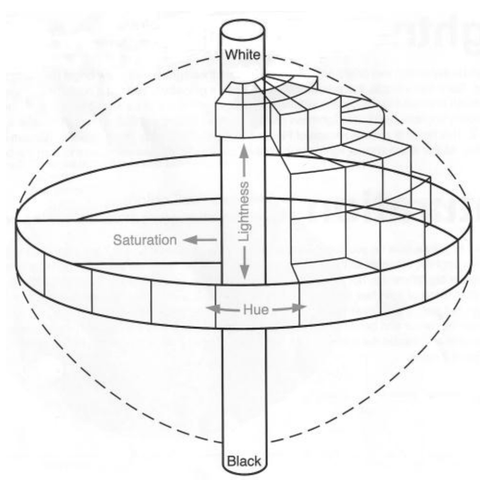

Figure 5.3. Three Dimensional Solid Using Hue, Lightness, and Saturation. Precise Color Communication: Color Control from Perception to Instrumentation. 14.

[63] Precise Color Communication: Color Control from Perception to Instrumentation. Konica Minolta. New Jersey: Konica Minolta Sensing, Inc, 2007. 12-15.

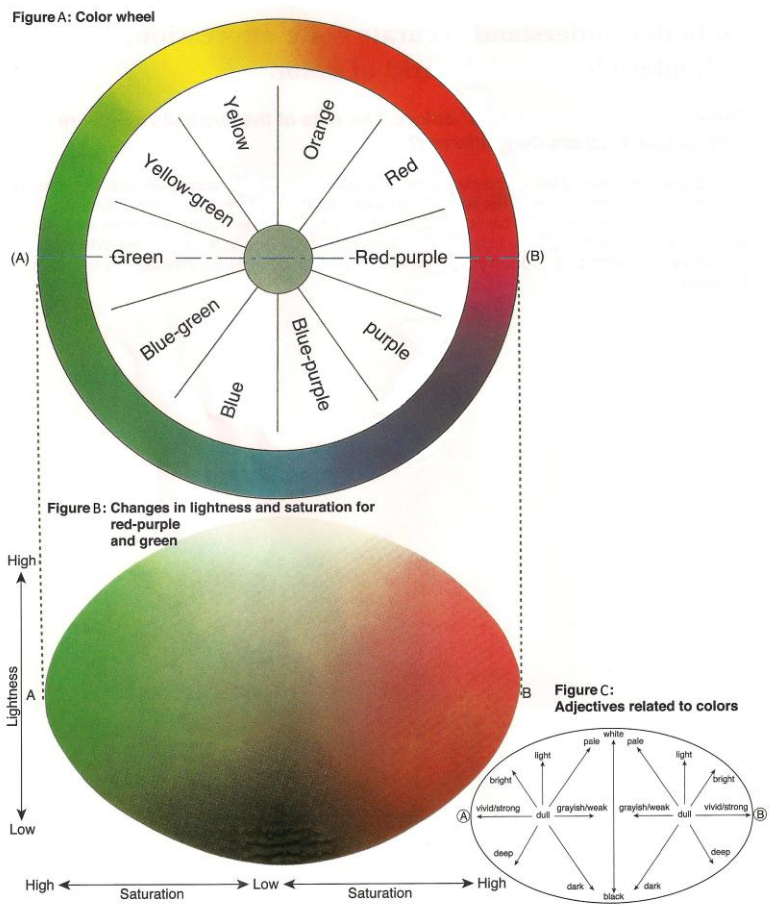

Figure 5.4. The Three Attributes of Color: Hue, Lightness, and Saturation.
Precise Color Communication: Color Control from Perception to Instrumentation. 12.

Through the combination of these three attributes a solid color is created. It is these three attributes that are measured using a colorimeter to determine exactly what combination of hue, lightness, and saturation make up a color, thereby quantifying a color. It is also through these number measurements that change in color can be measured and quantified.

A Minolta Chroma meter CR-400 was used and calibrated using a Minolta Calibration Plate CR-200/CR-300/CR400 and was set for 2 degree Observer. Each sample was measured 5 times and averaged. The colorimeter was placed in the middle of each sample in order to ensure consistency each time the test was performed so that the measurements were taken from the same spot on the sample each time. The data were taken in CIE, a*, b* coordinates for the purpose of documenting color and color change on the surface of the samples.

The colorimeter measures three components of light: lightness (L*), red-green value (a*), and yellow-blue value (b*). The coordinates of the color sphere are defined as CIE color space, L* a* and b*. The total color difference, ΔE^*, can be calculated from:

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]1/2$$

This equation can be used to determine the total color difference between two Lab color measurements; however, it does not tell in what way the colors differ. ΔL^* is the lightness value difference between sample color 1 and color 2; $\Delta L^* = L^*1 - L^*2$. Δa^* is the red-green value difference between sample color 1 and color 2; $\Delta a^* = a^*1 - a^*2$. Finally, Δb^* is the yellow-blue value difference between sample color 1 and color 2; $\Delta b^* = b^*1 - b^*2$.

<u>Laser Profilometer</u>

Changes in surface texture were observed using a Laser Profilometer. Both surface deterioration and accumulation of salt on the sample surfaces were monitored using this instrument to determine the extent of change in the surfaces of each material.

Surface texture is the local variation in the surface of an object from its ideal shape. Laser profilometry uses optical triangulation by using a light source (in this case a laser), imaging optics, and a photodetector. "The laser is focused on to the surface of the sample. Reflected light is focused on to the photodetector, which generates a signal that is proportional to the position of the spot in its image plane. As the distance to the target surface changes, the imaged spot shifts due to parallax. To generate a three-dimensional image of the [sample] surface, the sensor is scanned in two dimensions, thus generating a set of distance data that represents the surface topography of the [sample]."[64]

Surface texture can be characterized by a number of different variables defined by international standards. Some of the variables are as follows:

Sa – arithmetic mean deviation of the surface
Sp – highest peak surface, the height of the highest peak in
the roughness profile over the evaluation area
Sv – valley depth from surface
St – total height of the surface, the sum of Sp + Sv
Sku – kurtosis of the surface height distribution
Svk – reduced valley depth
Sk – core roughness depth
Spk – reduced summit height of the surface
Sfd – fractal dimension of the surface (complexity of the surface)
Sq – root mean square of the roughness
Vv – void volume of the valleys

A Solarius LaserScan was used throughout this experiment. It is a 3-D non-contact laser profilometer that uses a class II diode laser (670 nm wavelength) and a 2 µm spot size. The vertical resolution of the instrument is 0.1 µm and the maximum vertical range is 1 mm. This range allows observation of the peaks and valleys on stone surfaces. The laser scans over an area of 31.07 mm (x-axis) by 23.02 mm (y-axis) at a scan speed of 5 mm/s and a resolution of 25 µm. The estimated run time per sample is 111 minutes.

The samples had a notch etched into the edge of them. When placed in the laser profilometer this notch always lined up with a corresponding line on the instrument's sample holder. In this way, the sample was always in the same precise position when its surface was being scanned. This ensured that the same area of each sample was scanned and recorded each time.

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[64] Church, Jason. "Thesis." Message to cmoshida2@gmail.com. 10 Aug 2011. E-mail.

X-Ray Diffraction

A Shimadzu X-Ray Diffractometer XRD-6000 (Figure 7.3) was used for the X-Ray Diffraction test that was performed on certain samples at the beginning and the end of the experiment (Figure 5.5). The addition of a chemical compound to the sample material could cause a rearrangement of atoms and chemical bonds, thus changing the chemical structure of the material. The XRD identified residual or new materials formed upon treatment and weathering. The materials were identified based on their ability to diffract X-rays in order to measure interplanar spacing. Crystalline structures have unique X-ray diffraction patterns.

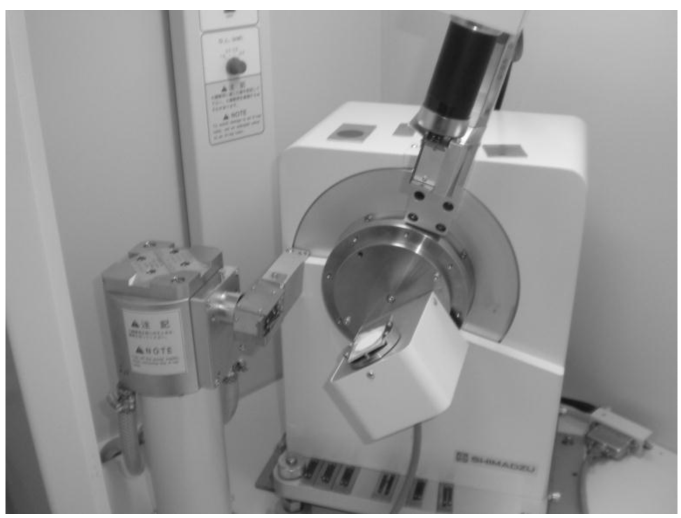


Figure 5.5. Non-Treated Limestone Sample in the XRD.

The X-ray tube is copper (Cu; 1.54060 A). The voltage was set at 40.0 kilovolts (kV) and the current was 30.0 milliampere (mA). This is a destructive test. In order to perform this experiment, the sample materials must be grinded down to a fine powder. A hand grinder was used to grind samples of the masonry material.

Fourier Transform Infrared Spectroscopy (FT-IR)

Fourier Transform Infrared Spectroscopy (FT-IR) is used to identify organic and inorganic chemicals in solids, liquids, and gasses. “In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radition is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample.”[65]

A PerkinElmer Spectrum One FT-IR Spectrometer was used to determine if chemical changes occured on the surface of each of the samples. The computer program used was PerkinElmer Specturm. The start (cm^{-1}) was set for 4000, the end (cm^{-1}) was set for 400, and the accumulations were 16 scans. The absorbance of each of the samples could be compared and changes in the materials’ spectra were observed.

Absorption – Rilem Tubes

The rate at which a material absorbs deionized water will help estimate the rate of absorption and the readiness of the material to absorb other possible other liquids such as herbicides. Rilem tubes were used to measure the amount of DI water each sample absorbed over a period of time. Each tube was attached to the sample using clay and filled with 7.5 mL of DI water (Figure 5.6).

[65] "Introduction to Fourier Transform Infrared Spectrometry." *Thermo Nicolet* 2. Web. 27 Aug 2011. <http://mmrc.caltech.edu/FTIR/FTIRintro.pdf>.

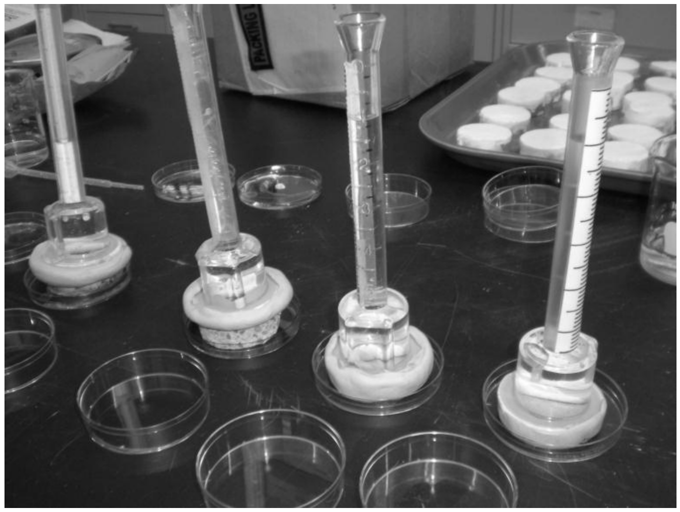


Figure 5.6. Rilem Tubes Filled with DI Water Attached to Concrete Samples.

Measurements of how much water the material absorbed were recorded at 5 minutes, 10 minutes, 15 minutes, 20 minutes, 30 minutes, and 60 minutes per the American Society for Testing and Materials (ASTM) guidelines for absorption testing on masonry. From these measurements, the rate of absorption of each sample was calculated and compared, which demonstrated increases or decreases in sample surface pore size.

<u>Masonry Materials: Brick, Concrete, Limestone, and Granite</u>

Forty-eight individual samples were used in this experiment; twelve of each stone/masonry material (see Chapter 3 for preparation of samples). Three samples of each were not exposed to any treatment solution and acted as the base/original controls for this experiment. The other nine samples were exposed to solutions in the following manner: 3 Roundup®, 3 Garlon®4, and 3 deionized water.

The identification used for each of the samples was the following. The first letter represents the type of treatment that the sample was exposed to: 'R' for Roundup®, 'G' for Garlon®4 and 'W' for deionized water. 'N' denoted that no treatment was used. The second letter signified the type of material it was: 'B' for brick, 'L' for limestone, 'C' for concrete, and 'G' for granite. The number depicted which of the 3 replicate samples of that herbicide/material combination it was. The identification labels were as follows:

NB1 = No treatment, brick, sample 1
NB2 = No treatment, brick, sample 2
NB3 = No treatment, brick, sample 3
NL1 = No treatment, limestone, sample 1
NL2 = No treatment, limestone, sample 2
NL3 = No treatment, limestone, sample 3
NC1 = No treatment, concrete, sample 1
NC2 = No treatment, concrete, sample 2
NC3 = No treatment, concrete, sample 3
NG1 = No treatment, granite, sample 1
NG2 = No treatment, granite, sample 2
NG3 = No treatment, granite, sample 3
RB1 = Roundup®, brick, sample 1
RB2 = Roundup®, brick, sample 2
RB3 = Roundup®, brick, sample 3
RL1 = Roundup®, limestone, sample 1
RL2 = Roundup®, limestone, sample 2
RL3 = Roundup®, limestone, sample 3
RC1 = Roundup®, concrete, sample 1
RC2 = Roundup®, concrete, sample 2
RC3 = Roundup®, concrete, sample 3
RG1 = Roundup®, granite, sample 1
RG2 = Roundup®, granite, sample 2
RG3 = Roundup®, granite, sample 3
GB1 = Garlon®4, brick, sample 1
GB2 = Garlon®4, brick, sample 2
GB3 = Garlon®4, brick, sample 3
GL1 = Garlon®4, limestone, sample 1
GL2 = Garlon®4, limestone, sample 2
GL3 = Garlon®4, limestone, sample 3
GC1 = Garlon®4, concrete, sample 1
GC2 = Garlon®4, concrete, sample 2
GC3 = Garlon®4, concrete, sample 3
GG1 = Garlon®4, granite, sample 1
GG2 = Garlon®4, granite, sample 2
GG3 = Garlon®4, granite, sample 3
WB1 = Deionized water, brick, sample 1
WB2 = Deionized water, brick, sample 2
WB3 = Deionized water, brick, sample 3
WL1 = Deionized water, limestone, sample 1
WL2 = Deionized water, limestone, sample 2
WL3 = Deionized water, limestone, sample 3
WC1 = Deionized water, concrete, sample 1
WC2 = Deionized water, concrete, sample 2
WC3 = Deionized water, concrete, sample 3
WG1 = Deionized water, granite, sample 1
WG2 = Deionized water, granite, sample 2
WG3 = Deionized water, granite, sample 3

All ‘N’ samples were not treated nor were any of them placed inside the QUV. They were virgin samples, strictly for the purpose of providing information about each of the masonry samples prior to any treatment conducted in this experiment. The other samples had the following tests performed on them before treatment, at a midpoint during the QUV cycle (337 hours), and after 800 hours in the QUV: weight, pH, and color. All samples (except for those with an ‘N’) containing the number ‘1’ were placed in the Laser Profilometer before treatment, at a midpoint during the QUV cycle, and after 800 hours in the QUV to map the surface of the material. These samples, after having their surfaces mapped, then underwent an absorption test using Rilem tubes. At the conclusion of the experiment, samples with the number ‘2’ (except for ‘N’ samples) were used for the X-RD test for comparison to the original, non-treated samples. However, prior to their destruction for that test, these samples were placed in the FT-IR machine for comparison to the original samples.

CHAPTER 6

EXPERIMENTAL PROCEDURE

Prior to treatment, all samples were individually placed under the camera with an identifying card and photographed. For each photograph, the camera was placed 32.385 centimeters above the table surface where the sample was resting. The Eiko Supreme Photoflood (ECT 120 volts) lights were the only source of illumination during photo documentation, and the lights were always positioned in their identical full upright position.

The mass of each sample was also taken. The scale was tared to 0.000 grams and the sample placed on the scale. The mass of the sample was recorded, the sample was removed, and the scale was tared to 0.000 g again. Each sample went through this routine 3 times; getting its mass measured a total of 3 times. The average of the 3 measurements was taken and recorded as the mass of the sample.

The Minolta Chroma meter CR-400 was used to measure the color if each sample. It was calibrated using a Minolta Calibration Plate CR-200/CR-300/CR400 and set for 2 degree Observer. For consistency, the middle of each sample was used. Each sample was measured 5 times and the average recorded.

Litmus paper was used to perform the pH test. The litmus paper was placed on the surface, preferably a side surface, of the masonry material. Deionized water (pH of DI water was a neutral 7) was used to wet the paper, which then adhered it to the sample. The litmus paper was left in contact with the sample for at least 1 minute. The litmus paper color was then determined, recorded, and both the sample and the used litmus paper were photographed.

Samples with the number '1' were placed in the laser profilometer for their surface profiles to be documented. The notch in each sample was aligned with the line on the sample holder to ensure that the same sample area was scanned each time throughout the experiment. Four samples could be measured at once. The samples had to be located at approximately the same height in the holders. Small pieces of paper were placed under some samples to raise them up to the required height.

Samples with the number '1' were also used to determine the absorption rate of the materials. Rilem tubes were used to determine absorption rate. Each tube was attached to the sample using clay and filled with 7.5 mL of DI water. Measurement readings of how much water the material was absorbing were recorded at 5, 10, 15, 20, 30, and 60 minutes. From these measurements the rate of DI water absorption of each sample was calculated. Before treatment, the absorption test was run in the NCPTT Laboratory. The room temperature was 72 degrees Fahrenheit and the relative humidity was 50 percent.

The FT-IR was performed on an untreated sample of each of the masonry materials (NB1, NL1, NC1, NG1). The PerkinElmer FT-IR Spectrum One Spectrometer was set at the following parameters:

- Methodology used: Attenuated Total Reflection (ATR)
- Abscissa Units = Wave number
- Ordinate Units = %T
- Start (cm-1) = 4000
- End (cm-1) = 400
- Resolution (cm-1) = 16
- Accumulations = 16 scans

The results were saved as the baseline for later comparisons.

For the X-Ray Diffraction test, extra samples that were not treated with any herbicides or DI water were used as baseline comparison samples. The samples had to be grinded into powder using a hand grinder (Figure 7.1). Between samples, the grinder was cleaned thoroughly so as not to contaminate samples.

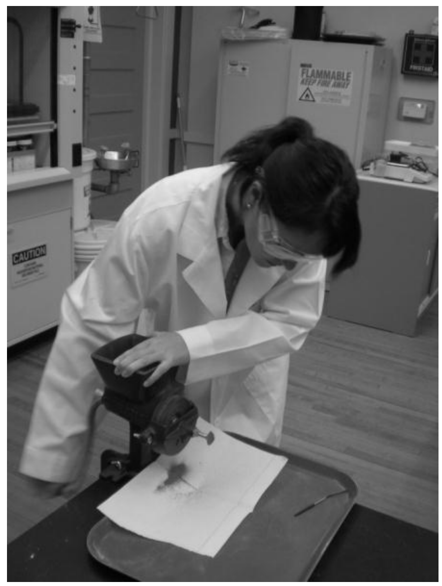


Figure 6.1. Using a Hand Grinder to Turn a Brick Sample into a Powder to be Tested Using the XRD.

The step-size for all tests was 0.0200 degree. The non-treated brick sample was run at 2 degrees per minute rotation speed with angles 10.0 to 80.0 degrees. The non-treated limestone sample was run at 2 degrees per minute rotation speed with angles at 10.0 to 80.0 degrees. The non-treated cement sample ran at 2 degrees per minute rotation speed with angles at 10.0 to 40.0 degrees. The non-treated granite sample ran at 2 degrees per minute rotation speed with angles 10.0 to 40.0 degrees. Each stage had to be adjusted so that the material remained flat and smooth inside the container; unevenness could cause misreadings and calculation errors which would affect the results.

0 Hours to 337 Hours

On December 20, 2010, all the samples were treated in their sample holders with their designated solution according to the color of tape mounted on the back of the QUV racks: green for Roundup®, orange for Garlon®4 or blue for deionized (DI) water. They were then placed in the QUV and the cycles begun. The QUV was programmed to maintain a temperature of 50 degrees Celsius and run on 4 hour cycles, alternating from dry and wet.

At 268 hours, the samples were removed from the QUV and re-sprayed twice with their appropriate solution. The samples were tightened in the holder and made secure as some came loose during the QUV condensation wet/dry cycles. They were then placed back into the QUV to continue the experiment.

337 Hours to 800 Hours

The QUV was paused and the samples removed from the QUV at 337 hours, 2 hours and 21 minutes into UV1 cycle on January 6, 2011. Each sample was placed on a tray that had pins sticking into it in order to elevate the sample and to prevent either face of the masonry material from touching the tray and possibly removing any salts or evidence of change. The trays with the samples were then placed in an oven at 70-75 degrees Celsius for 1 hour to evaporate the water and dry them for testing. Sample GG2 still appeared wet after 1 hour, so it was placed back into the oven for an additional 3 hours until all the liquid on it had evaporated.

Before sample testing began, observations (color, presence/absence of efflorescence) were made regarding the appearance and each sample photographed (Chapter 7).

The tests for weight, color, pH, and surface profile were then performed (only on samples with the number “1”). The tests were performed exactly as they were prior to treatment. The samples were then placed back into the holders that were designated for their type of treatment and re-sprayed with their specified solution. They were placed in the QUV and the cycle restarted.

On January 11, 2011, it was discovered that sample GG1 fell out of the QUV sample holder and lay in the condensate tray of water for an unknown amount of time approximately between the hours of 5:00 PM and 7:30 AM. Upon discovery, the sample was picked up and placed back in its holder.

On January 13, 2011, at 7:30 AM, it was found that the QUV had not run all night due to a low temperature warning. Also, sample WL1 had fallen out and was found lying in the condensate pan. The sample was placed back into its holder and the QUV was restarted.

On January 24, 2011, at 2:20 PM, the QUV was stopped in the final minutes of the UV cycle, at 635 hours. All the samples were then re-sprayed with their designated herbicide solutions, and placed back into the QUV. The QUV was then restarted.

<u>800 Hours</u>

On February 1, 2011, the QUV was turned off when it reached 800 hours at about 11:24 AM. All the doors on the QUV were then opened to allow the samples to dry, and on February 7, 2011 they were removed from the QUV and placed on the trays with the pins for testing.

Testing began March 10, 2011. Again all samples were tested the exact same way as done prior to treatment and at the midway point (337 hours). All the samples were photographed. The weight of each sample was taken, the color determined using the colorimeter, and the pH level measured using litmus paper. All samples with the number "1" in their identification sequence were placed in the laser profilometer to measure and document surface texture. Afterwards, these samples were tested for absorption rates using Rilem tubes. The test was conducted in the NCPTT Laboratory where the room temperature was 75 degrees Fahrenheit and the relative humidity was 50 percent. Meanwhile, samples RB2, RL2, RC2, RG2, GB2, GL2, GC2, GG2, WB2, WL2, WC2, and WG2 were each placed in the FT-IR for testing (the settings were the same as at 0 hours for the non-treated samples). Afterwards a comparison of each sample with a control (baseline) sample (non-treated) and a control sample (sprayed with DI water) were plotted on the same graph (Appendix G). Two of the sample traces were compared using the Spectrum10 software from PerkinElmer and a difference (the source subtracted from the control) resulted, providing enough data for the identification of the two herbicides used.

Finally, samples with the identification number "2" were ground into a fine powder using the hand grinder and placed in the X-RD for testing on October 4, 2011 by Jason Church, NCPTT. All samples had a scan range of 10-80 degrees, a step size of 0.0200 degree, and the rotational stage was at 20 rpm. The Slit DS was 1.00 degree, the SS was 1.00 degree and the RS was 0.30 mm. An overlaid spectrum of each of the same masonry material (one treated with Roundup®, one treated with Garlon®4, and the control sample of DI water) were plotted for comparison.

CHAPTER 7

RESULTS

Throughout the experiment, different tests and data collection methods were performed to document the physical and chemical changes in each of the samples.

Observations at 337 Hours

The following observations were made at the midpoint (337 hours) of the QUV session:

- Sample RB2 had efflorescence present on the front face of the sample upon removal from the QUV.
- Efflorescence was also present on the back faces of samples GC1, GC2 and GC3.
- All of the DI water samples had no visible change in appearance. They all looked as they did before they were placed in the QUV.
- Limestone and granite samples that had been treated with Garlon®4 herbicide were covered in an orange stain, making them appear darker than the original coloring and slightly more orange.

The rest of the chapter focuses on the results taken from the tests from the beginning of the experiment until the end (800 hours in the QUV).

Photographs

Photographs were taken to document visible changes to the samples throughout the experiment process such as salt/efflorescence formation and color change. All photographs are in Appendix B.

In photographs documenting RB1, RB2 and RB3 samples, the appearance of a whitish haze is visible in the after 800 hour photographs. This discoloration is the formation of salt/efflorescence on the masonry material after being exposed to Roundup® herbicide. This salt is also visible in photographs of GG2 and GG3. Although difficult to observe in the photographs, salt formations occurred on Roundup® limestone samples as a translucent haze, making the surface look glossy. The salt formation on the Roundup® concrete samples formed in a different location. The salt built upward in crevices rather than covering the entire surface of the material, which made it more challenging to photograph from straight above.

All samples that were treated with Garlon®4 were discolored at the completion of the experiment. Brick, limestone, concrete, and granite samples were all darker in color than they were originally (apparent in the photos). This was possibly caused by the orange dye put in the herbicide by the manufacturer.

Visual Survey Results

The photos taken (Appendix B) over the 800 hour test period were used in a survey that asked people to quantify the amount of change that they observed in each sample. A graduated rating system was used, with 1 being no change and 5 being the most physical change based on the photographs taken during the experiment. The people were asked what physical changes they observed: discoloration, efflorescence/appearance of white coloration, pits/holes, or other. Twenty-six surveys were completed in the allotted time, taken by people of varying ages (all over the age of 18) and backgrounds. The average ratings were as follows:

RB1 = 2.8	GB1 = 3.8	WB1 = 1.8
RB2 = 2.0	GB2 = 3.6	WB2 = 1.1
RB3 = 2.0	GB3 = 2.8	WB3 = 1.6
RL1 = 0.7	GL1 = 2.9	WL1 = 1.0
RL2 = 0.8	GL2 = 3.0	WL2 = 1.0
RL3 = 0.8	GL3 = 3.0	WL3 = 0.7
RC1 = 1.2	GC1 = 2.7	WC1 = 1.2
RC2 = 0.9	GC2 = 2.8	WC2 = 0.8
RC3 = 1.0	GC3 = 2.8	WC3 = 0.8
RG1 = 1.0	GG1 = 3.8	WG1 = 1.2
RG2 = 1.1	GG2 = 4.1	WG2 = 0.8
RG3 = 0.8	GG3 = 3.8	WG3 = 1.2

One hundred percent of the people surveyed observed discoloration in samples GB1, GB2, GB3, GL1, GL2, GL3, GG1, GG2, and GG3. Ninety-two percent saw discoloration in GC1 and GC2, while 96 percent saw discoloration in GC3. One hundred percent saw efflorescence in RB1, while 88 percent saw it on sample RB3. A large percentage of people also saw efflorescence on samples GG2 and GG3, however, upon closer inspection of the photograph, what people mistook as salt formation is actually the color of the original material showing. It appears white due to the rest of the surface being stained a darker color because of the exposure to Garlon®4 herbicide. Pits and holes were seen by a few people in some of the photographs. Fifty percent of the people saw pits and holes in sample RB2 and 27 percent saw pitting in GB3 (Figure 7.1, Appendix C).

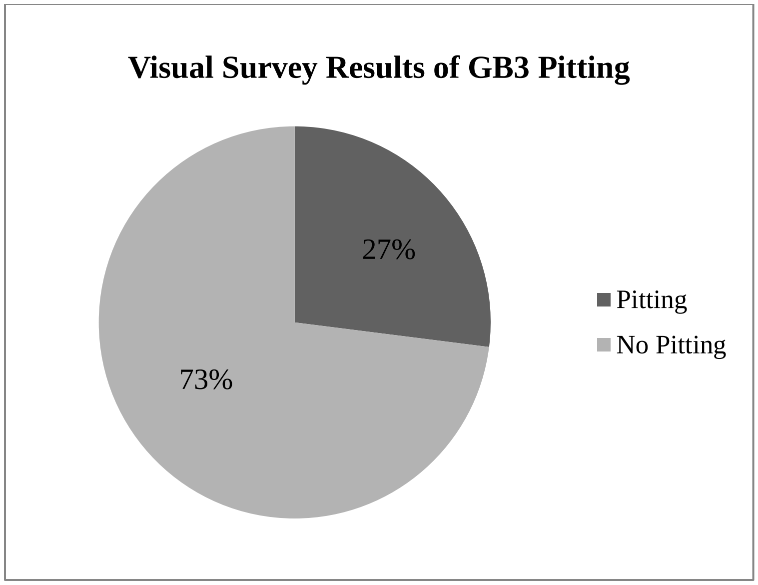


Figure 7.1. Visual Survey Results of GB3. Visual survey after treatment results for brick sample number 3 that was treated with Garlon®4. This shows that 27 percent of the people saw pitting on this sample, or at least an increase of surface roughness.

Overall, the survey helped determined if physical changes occurred to the samples after exposure to an herbicide solution and were noticeable to the public.

Mass Results

The mass of each sample was taken before undergoing any treatment, at 337 hours into the experiment (in the QUV), and at the conclusion of the experiment at 800 hours. At each weighing, each sample was weighed three times and the average of those measurements was calculated to be the weight of the sample at that time (Figure 7.1, Appendix H).

Most of the samples that were exposed and treated with an herbicide increased in mass. This could be because of the masonry materials' ability to absorb the chemicals and retain them for long periods of time based on their chemical composition. However, it is more likely that the smaller increases in mass were due to the excess buildup of salt/efflorescence on and in the material itself. The extra salt formation would increase the mass of the sample.

Overall, the Garlon®4 samples gained the most weight, with the brick samples gaining the most in the entire experiment followed by the limestone samples. The three brick samples that were treated with Garlon®4, with an average starting weight of 23.61 g, showed an average weight gain of 2.24 g. The next closest weight gain was by the Garlon®4 limestone samples (with an average starting weight of 15.90 g) with an average total weight gain of 0.90 g.

Samples RC2 and RC3 lost weight at the beginning of the experiment. This could have been due to loss of material during handling between experiments and testing. However, during the second interval in the QUV they both gained weight, most likely due to the buildup of salt on their surfaces, as is the case for most of the samples that came into contact with herbicide, but they are still lighter than at 0 hours.

The control samples (deionized water) had very little change in mass, which can be accounted for either by excess dirt/dust or other particles that had been left on the sample after cleaning, or small pieces of the masonry material might have been broken off at some point during the first 337 hours in the QUV or the materials ability to hold and retain moisture from the condensation cycles in the QUV.

Table 7.1. Average Sample Mass. The average mass in grams (g) of each sample was determined at each stage of the experiment.

Sample	Average Mass (g)		
	Before Treatment; 0 Hours	During Treatment; 337 Hours	After Treatment; 800 Hours
RB1	22.88	22.24	22.33
RB2	22.11	22.19	22.31
RB3	26.34	26.35	26.47
RL1	16.42	16.45	16.48
RL2	15.64	15.68	15.71
RL3	15.59	15.63	15.66
RC1	45.37	45.01	45.13
RC2	44.12	43.82	43.96
RC3	46.31	45.99	46.12
RG1	30.42	30.42	30.43
RG2	35.23	35.23	35.24
RG3	28.96	28.96	28.98
GB1	24.95	25.77	27.43
GB2	23.65	24.47	25.27
GB3	22.22	23.33	24.84
GL1	16.19	16.77	17.14
GL2	15.70	16.21	16.52
GL3	15.81	16.19	16.73
GC1	46.93	47.19	47.57
GC2	43.99	44.12	44.57
GC3	44.67	44.73	45.17
GG1	29.49	29.53	29.55
GG2	32.32	UNKNOWN*	32.39
GG3	29.26	29.32	29.32
WB1	24.60	24.39	24.41
WB2	21.87	21.86	21.86
WB3	27.60	27.58	27.61
WL1	15.99	15.98	15.98
WL2	15.89	15.89	15.89
WL3	15.08	15.08	15.08
WC1	43.52	43.25	43.34
WC2	44.81	44.46	44.58
WC3	43.63	43.29	43.41
WG1	32.87	32.86	32.86
WG2	32.05	32.05	32.05
WG3	30.07	30.07	30.07

*Misplaced during testing and not found.

pH Results

The litmus paper was tested to make sure it was still functional and reliable prior to use in this experiment. The pH paper has limited accuracy and precision, but it can easily be used to test pH on the surface of samples. The pH of each sample was tested before treatment, at 337 hours in the QUV, and upon the experiment's completion at 800 hours (Table 7.2).

All Roundup®-brick and Roundup®-limestone samples (RB1, RB2, RB3, RL1, RL2, RL3) increased in pH from the beginning of the experiment to the end. This shows that brick and limestone, upon exposure to Roundup®, became less acidic in chemical composition. In contrast, Roundup®-concrete samples had a slight decrease in alkalinity. Roundup®-granite samples showed very little pH change, with only RG3 increasing in pH.

Garlon®4-brick and Garlon®4-limestone samples (GB1, GB2, GB3, GL1, GL2, GL3) increased in pH after exposure to the herbicide. Sample GG3 also increased in pH. Sample GC1 decreased in pH. The rest of the samples remained in the pH 6-7 range with little or no change in their pH.

Deionized water-brick and deionized water-limestone samples (WB1, WB2, WB3, WL1, WL2, WL3) increased in pH after being treated with DI water, changing from readings of pH 5/6 to pH 6/7. Samples WC1, WC3 and WG2 also increased in pH, becoming more neutral. Samples WG1 and WG3 both decreased in pH from pH 7 to a more acidic pH 6, and sample WC2 remained constant at pH 7

Table 7.2. pH Results. The change in pH of each of the samples at each stage of the experiment.

Sample	**pH**		
	Before Treatment; 0 Hours	**During Treatment; 337 Hours**	**After Treatment; 800 Hours**
RB1	5	6	6
RB2	5	7	7
RB3	5	7	6
RL1	6	7	7
RL2	5	7	7
RL3	6	7	7
RC1	7 (with 8 spots)	7	7
RC2	8	7	7
RC3	7 (with 8 spots)	8	7
RG1	7	6	7
RG2	7	7	7
RG3	7	6	6
GB1	5	6	6
GB2	5	6	6
GB3	5	6	6
GL1	6	7	8
GL2	6	6	7
GL3	6	6	7
GC1	7 (with 8 spots)	6	6
GC2	7	7	7
GC3	7	7	7
GG1	7	7	7
GG2	7	6	7
GG3	6	6	7
WB1	5	6	6
WB2	5	6	6
WB3	5	6	6
WL1	6	7	7
WL2	6	7	7
WL3	6	7	7
WC1	7	8	8
WC2	7 (with 8 spots)	8	7 (with 8 spots)
WC3	7	8	8
WG1	7	7	6
WG2	6	7	7
WG3	7	7	6

Colorimeter Results

Colorimetry is a test that gives a numerical value to a color in order to be used to quantitatively compare colors. These values can also be used to compare the initial color of a sample to the color of the sample after treatment. All samples had their top surface color tested before treatment, at 337 hours in the QUV, and at the completion of the experiment (800 hours) (Appendix D). L* indicates lightness, a* indicates red-green colors, and b* indicates yellow-blue coloration. The overall color change is expressed as ΔE* (Table 7.3)

The samples that were treated with Garlon®4-herbicide had the largest amount of color change (Figure 7.2). The Garlon®4-granite samples (GG1, GG2, GG3) averaged an overall color change of 19.23, the greatest degree of change out of any of the sample combinations of masonry material and treatment. This was followed by Garlon®4-limestone samples (GL1, GL2, GL3), which averaged 17.88 degree of changes in the color of the material, and Garlon®4-brick samples (GB1, GB2, GB3), which averaged a change in color of 12.54. However, the greatest change for any single sample was GG1, which had a 20.06 degree of color change amount.

In contrast, the Roundup®-masonry material samples had significantly lower color change averages ranging from 1.20 (Roundup®-granite) to 5.21 (Roundup®-concrete). The overall average change of color of all samples that were treated with Roundup® is 3.03. The greatest amount of color change measured in any of the Roundup® samples is RC1 with a color change of 6.96, and the least amount of color change measured is GC1 with a change of 5.28.

The control treatment, deionized water samples also showed a low amount of color change in comparison to the samples treated with Garlon®4. The average change of all the deionized water samples is 2.79. The color changes of each combination of deionized water treatment and masonry material ranged from a color change of 0.52 (DI water-granite) to 4.37 (DI water-granite).

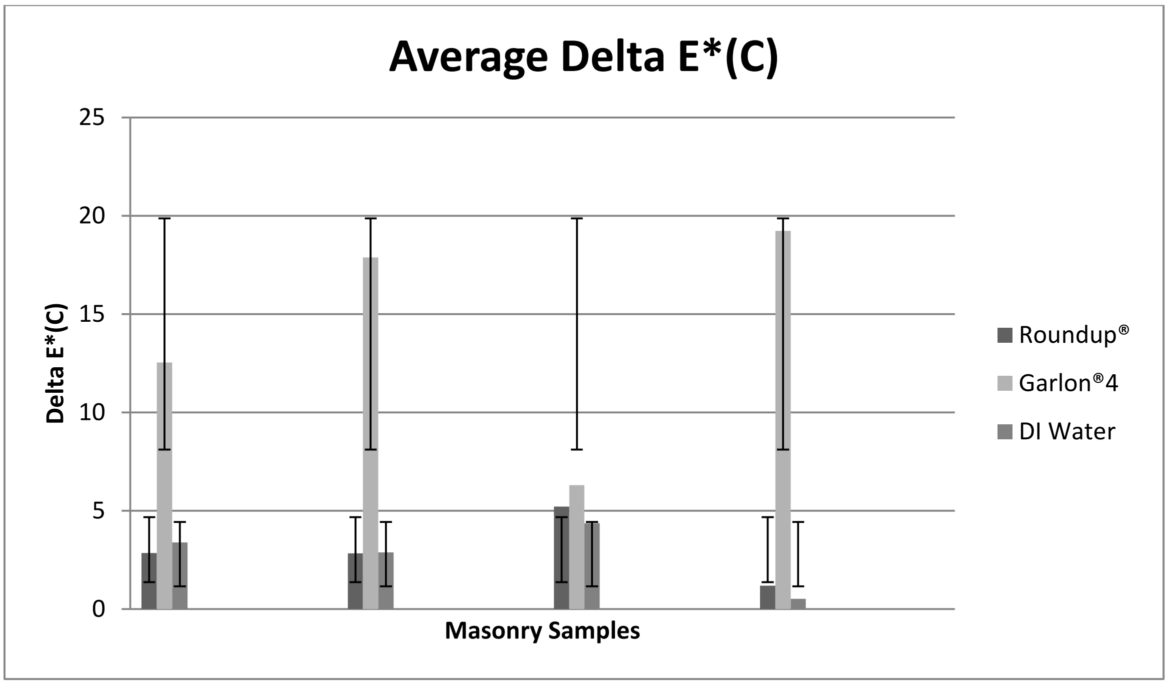


Figure 7.2. Average Change in the E*(C) of the Herbicide-Masonry Combinations. The first letter in the identification of the sample (x-axis) is the herbicide used: "R" is Roundup®, "G" is Garlon®4, and "W" is DI water. The graph shows that samples treated with Garlon®4 had the most color change occur during the experiment. Error bars with standard deviation.

Table 7.3. Change in the Colorimetry Measurements (800 Hours Measurements – 0 Hours Measurements).

	Data Name	L*(C)	a*(C)	b*(C)	E*(C)
1	RB1	3.57	-2.34	-3.92	5.80
2	RB2	0.88	-0.59	-1.82	2.11
3	RB3	0.02	0.19	-0.63	0.66
4	RL1	2.07	-0.45	-1.15	2.41
5	RL2	2.46	-0.54	-1.11	2.75
6	RL3	3.06	-0.57	-1.29	3.37
7	RC1	6.79	-0.15	1.51	6.96
8	RC2	4.90	0.98	1.00	5.25
9	RC3	3.37	0.63	0.07	3.43
10	RG1	1.86	-0.12	0.62	1.96
11	RG2	0.27	-0.26	-0.44	0.58
12	RG3	-0.72	-0.20	-0.75	1.06
13	GB1	-13.89	3.67	0.14	14.37
14	GB2	-9.67	6.06	1.54	11.52
15	GB3	-11.38	0.77	-2.70	11.72
16	GL1	-18.41	1.38	5.69	19.32
17	GL2	-16.08	0.97	5.06	16.89
18	GL3	-16.69	1.11	4.89	17.43
19	GC1	0.91	1.29	5.04	5.28
20	GC2	-3.83	1.23	4.37	5.94
21	GC3	-7.45	1.07	1.60	7.69
22	GG1	-14.53	0.61	13.81	20.06
23	GG2	-15.67	0.89	9.03	18.11
24	GG3	-14.50	0.68	13.06	19.53
25	WB1	4.67	0.87	1.66	5.03
26	WB2	0.14	-0.01	-0.17	0.22
27	WB3	4.93	0.30	0.12	4.94
28	WL1	2.55	-0.57	-1.10	2.84
29	WL2	2.49	-0.46	-0.88	2.68
30	WL3	2.88	-0.54	-1.12	3.14
31	WC1	5.07	0.18	0.28	5.08
32	WC2	4.56	0.16	0.75	4.62
33	WC3	2.71	1.43	1.49	3.41
34	WG1	-0.08	-0.16	-0.19	0.26
35	WG2	-1.12	-0.20	0.22	1.16
36	WG3	0.12	0.04	0.05	0.14

Laser Profilometer Results

Twelve samples of varyious treatment and masonry material had their surface texture mapped using a laser profilometer before, during, and at the conclusion of the experiment (Figure 7.3, Figure 7.4, Appendix E).

For this study, the reduced summit height of the surface/peaks (Spk) value and the reduced valley depth/valleys (Svk) value will be most helpful in indicting changes to the surface of the material. For each of the values, a baseline is established (Sk) and then the laser profilometer calculates the number of peaks above that baseline (Spk) or the number of valleys below that baseline (Svk). So, if there is a larger number for the Spk after treatment, there are more peaks, which means that the surface got rougher. The larger the number for the Svk means that there are more valleys, which means that there is more pitting occurring ("Swiss cheesing" of the material).

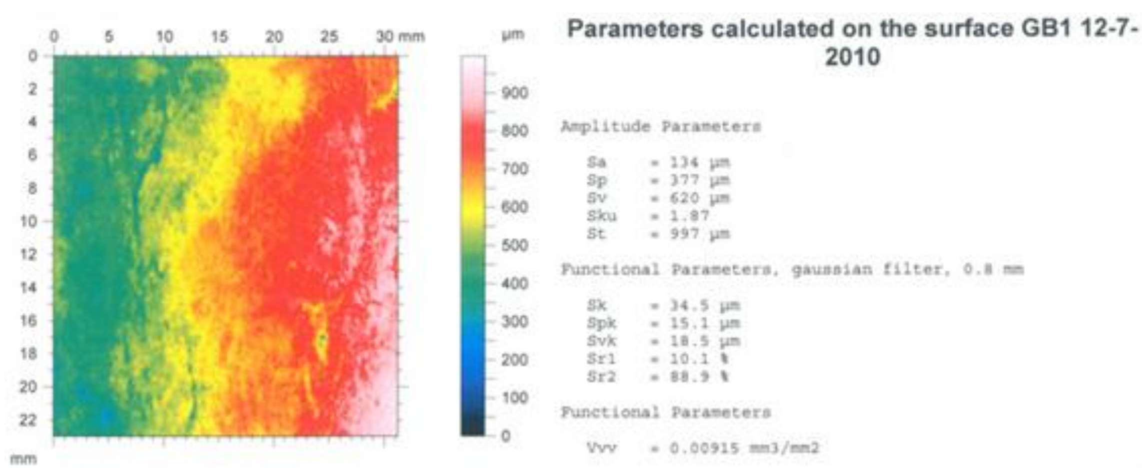


Figure 7.3. Laser Profilometer Measurements for Sample GB1 Before Treatment.

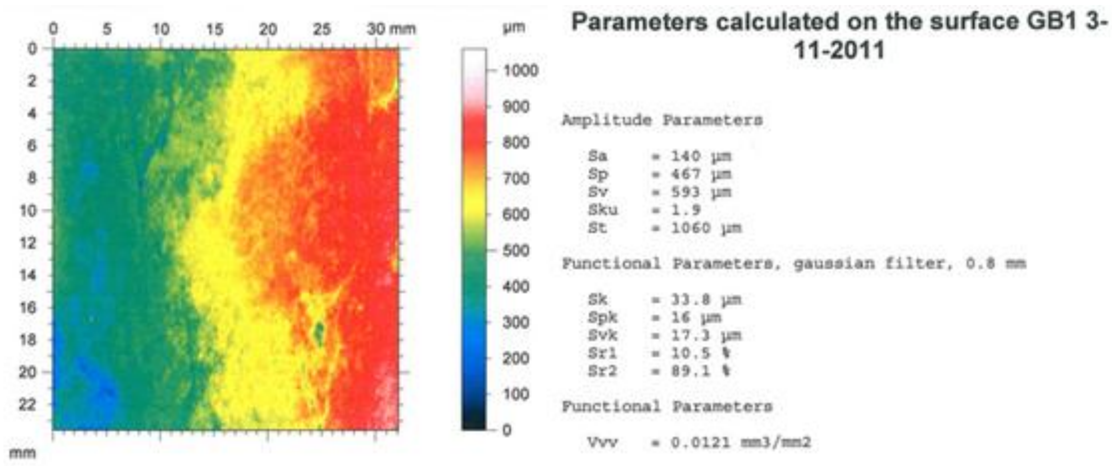


Figure 7.4. Laser Profilometer Measurements of Sample GB1 After 800 Hours in the QUV and 4 Exposures to Garlon®4 Herbicide. Notice the changes in color which depict changes in surface texture.

X-Ray Diffraction (XRD) Results

The X-ray diffractometer measures a material's atomic structure. Each crystalline structure has a unique X-ray diffraction pattern. A comparison of each masonry material with two treated samples (Roundup® sample and the Garlon®4 sample) and a control sample (DI water/non-treated sample) were plotted on a graph after analysis. (Appendix F) The main components in Garlon®4 are organic in nature and were not expected to show in XRD analysis. The main components in Roundup® are salt compounds that should be visible by XRD.

The overlaid spectrum of samples RB2, GB2, and WB2 showed no significant increase in compositional make up. The overlaid spectrum of samples RL2, GL2, and WL2 showed no significant increase in compositional make up. The overlaid spectrum of samples RC2, GC2, and WC2 showed no significant increase in compositional make up. Lastly, the overlaid spectrum of samples RG2, GG2, and WG2 also showed no significant increase in compositional make up.

Fourier Transform Infrared Spectroscopy (FT-IR) Results

By examining the chemical spectrum of the samples, it can be determined if there was any chemical residue left on the material after exposure to the herbicides by comparing the after exposure samples to the baseline samples that were untreated and control samples coated with DI water throughout the test. (Appendix G)

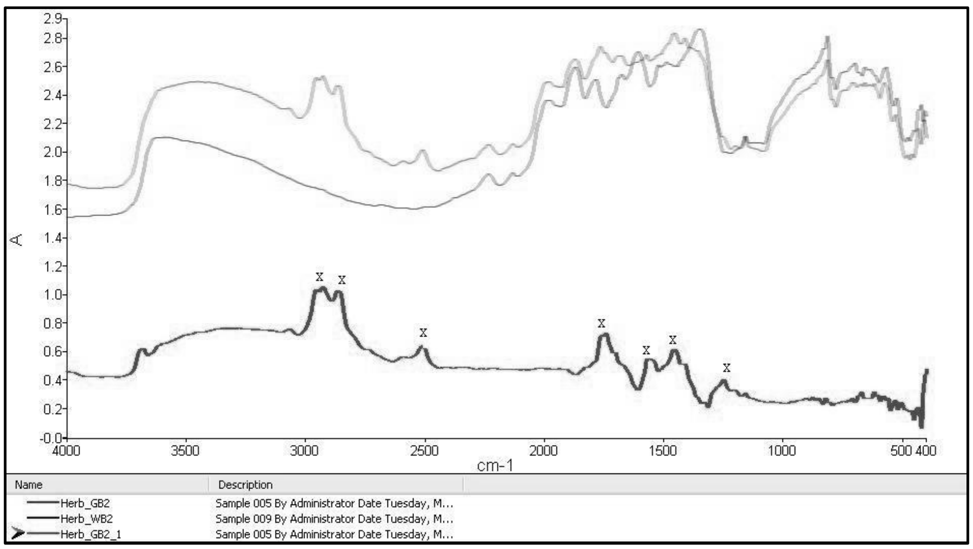


Figure 7.5. FT-IR Garlon®4 Results. The above FT-IR trace illustrates the Garlon®4 residue left after treatment. The (X) markers are the peaks associated with Garlon®4.

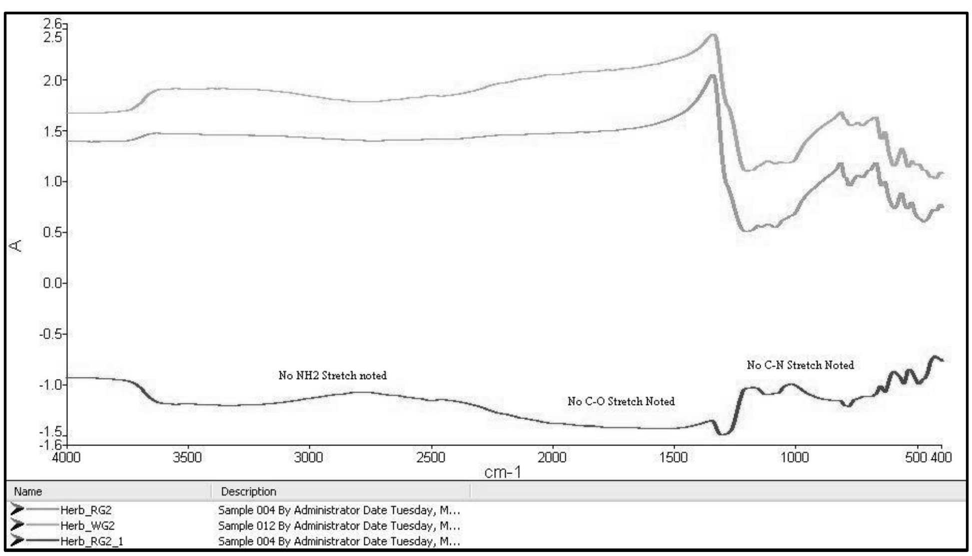


Figure 7.6. Trace Above Illustrates the Absence of Any Peaks That Would Identify Roundup®.

The two samples that were treated with herbicide per masonry material had their spectra compared using the Spectrum10 software from PerkinElmer and the difference (the source subtracted from the control) resulted in giving enough data for the identification of the two herbisides. Thus, when the spectra were examined, the correct peaks could be identified along the spectrum that correlated to each herbicide, and it could be determined if that herbicide was still present on the materials' surfaces. Also, by reading the list of ingredients and chemicals in each of the herbcides as given on their material safety data sheets (MSDS), a trained chemist (in this case Curtis Deselles, NCPTT) would know what specific peaks to look for and where on the spectrum to identify their presence. Mr. Deselles aided in the analysis process of identifying peaks within the spectrum of each sample material.

Roundup® was not identified on any of the masonry samples tested (Figure 7.5). However, Garlon®4 was found on the substrates of brick, concrete, and limestone (Figure 7.6). But it was not found on the granite samples.

Absorption Results

The rate at which the material absorbs deionized (DI) water helps determine the rate of absorption and the possible readiness of the material to absorb other liquids such as herbicides. If there was an increase in the rate of absorption over the same amount of time, it would indicate that the pores of the material are increasing either in number or size, enabling DI water to be absorbed at a faster rate. However, if the rate decreases, it would indicate that the pores are getting smaller or diminishing in number, possibly due to blockage by the herbicide either in salt form or oil residue.

Rilem tubes were used to determine absorption rate. Only the following samples were tested: RB1, RL1, RC1, RG1, GB1, GL1, GC1, GG1, WB1, WL1, WC1, and WG1. The following Tables (7.4 and 7.5) show the measurements recorded for the samples throughout the experiment.

Table 7.4. Absorption Results Before Treatment. The absorption readings taken from the Rilem tubes at the specified time before treatment (0 hours). The measurements record how much water the masonry material absorbed (mL) over a certain amount of time.

Sample	**Time in Minutes**						
	5	**10**	**15**	**20**	**30**	**60**	
RB1	2.3	2.6	3.0	3.3	3.9	5.2	**Absorption Rate of the material; how much each sample absorbed in mL**
GB1	5.5	6.0	6.0	6.0	6.0	6.0	
WB1	2.0	2.2	2.5	2.7	3.1	4.2	
RL1	0.2	0.4	0.4	0.5	0.5	0.7	
GL1	0.7	0.8	0.9	0.9	1.0	1.1	
WL1	0.6	0.7	0.8	0.8	0.9	1.0	
RC1	0.2	0.6	0.9	1.7	2.3	3.7	
GC1	0.2	0.2	0.2	0.2	0.3	0.3	
WC1	0.5	1.0	1.0	1.8	2.5	3.9	
RG1	0.0	0.0	0.0	0.0	0.0	0.0	
GG1	0.0	0.0	0.0	0.0	0.0	0.1	
WG1	0.0	0.0	0.0	0.1	0.1	0.1	

Table 7.5. Absorption Results After Treatment. The absorption readings taken from the Rilem tubes at the specified time after (800 hours). The measurements record how much water the masonry material absorbed (mL) over a certain amount of time.

Sample	**Time in Minutes**						
	5	**10**	**15**	**20**	**30**	**60**	
RB1	3.6	4.5	5.0	5.5	5.7	6.2	**Absorption Rate of the material; how much each sample absorbed in mL**
GB1	0.8	1.0	1.7	1.9	1.2	5.6	
WB1	3.0	3.5	3.8	4.2	4.6	5.7	
RL1	0.0	0.0	0.1	0.2	0.3	0.7	
GL1	1.1	2.3	3.7	4.9	5.5	6.5	
WL1	0.4	0.6	0.6	0.7	0.9	1.0	
RC1	0.1	0.2	0.4	0.6	1.2	3.0	
GC1	0.0	0.0	0.1	0.1	0.2	0.6	
WC1	0.2	0.3	0.4	0.4	0.5	0.8	
RG1	0.1	0.1	0.1	0.1	0.2	0.2	
GG1	0.0	0.0	0.0	0.0	0.0	0.0	
WG1	0.2	0.2	0.2	0.3	0.3	0.4	

Most of the masonry materials initially absorbed their maximum volume of the DI water within the first 10 minutes of the experiment. The rates at which the samples absorbed water decreased over the remainder of the experiment. The absorption rate is change over time, so it is a first order kinetic equation because simple exponential decay is first order. Because the majority of the water a masonry material sample can hold is absorbed within the first ten minutes of exposure, the main focus is at those points.

Initially, it seems that samples exposed to Roundup® had a faster rate of absorption after exposure while Garlon®4 samples had a slower rate of absorption after treatment. This could indicate the Roundup® is causing pores of the materials it contacts to widen or increase, while Garlon®4 is blocking or in some way inhibiting the passage of DI water into the material.

CHAPTER 8

DISCUSSION

This chapter will seek to find relationships between the herbicide and the masonry material through the analysis of the data gathered throughout the experiment. Both the physical and chemical changes, if any, will be examined and a conclusion drawn about each of the herbicides' effects on masonry materials.

Brick

The results from the brick samples will be examined, compared, and discussed.

Table 8.1. Results of Some of the Physical Tests on the Brick Samples.

Sample	**Herbicide**	**Salt Formation After 337 Hours QUV**	**Salt Formation After 800 Hours QUV**	**Δmass (in grams)**	**ΔSk (in mm)**	**ΔSpk (in mm)**	**ΔSvk (in mm)**
RB1	Roundup®	Yes	Yes	-0.55	0.0023	-0.0011	0.0007
RB2	Roundup®	Yes	Yes	0.2	---	---	---
RB3	Roundup®	No	Yes	0.13	---	---	---
GB1	Garlon®4	No	No	2.48	-0.0007	0.0009	-0.0012
GB2	Garlon®4	No	No	1.62	---	---	---
GB3	Garlon®4	No	No	2.62	---	---	---
WB1	Control	No	No	-0.19	0.0018	0.0005	0.0007
WB2	Control	No	No	-0.01	---	---	---
WB3	Control	No	No	0.01	---	---	---

As shown in Table 8.1, a white haze was seen on samples RB1 and RB2 after 337 hours in the QUV and 1 exposure to Roundup®, and on RB3 after 800 hours in the QUV and 2 treatments of Roundup®. The white haze is salt formation, or efflorescence. Compared to the control samples (WB1, WB2, WB3) that had no efflorescence on their surfaces after 337 hours or 800 hours, the appearance of efflorescence can be attributed to the exposure to Roundup® herbicide. The brick samples that were sprayed with Garlon®4 throughout this experiment (GB1, GB2, GB3) did not develop efflorescence.

The mass of brick samples that were sprayed with Roundup® had varying degrees of change throughout the experiment, as shown in Table 8.1. The small increase in weight shown by all three samples can be attributed to the salt build up in and on the surface of the material, which was visible. Samples RB2 and RB3 show that continual exposure to Roundup® increases the amount of efflorescence on the material and thus slightly increases the weight of the brick samples. All three brick-Garlon®4 samples increased in weight. This increase in weight could be due to the bricks' ability to absorb and retain solutions as there is no evidence of efflorescence in these samples. There were no dramatic increases or decreases in weight in the samples treated with DI water. The largest decrease in weight was sample WB1 which decreased in weight by 0.21 g during the first phase of the experiment. This may be attributed to the dirt that was lost from the surface of the material by the moisture while in the QUV.

As shown in Table 8.1, the Spk of sample RB1 decreased by 0.0011 mm. This shows that the number of peaks decreased from the beginning of the experiment, before treatment, to exposure to Roundup® and completion of the experiment. The Svk for this sample increased by 0.0007 mm showing that the number of valleys increased, which indicates that there is pitting occurring on the surface of the brick. The GB1sample that was exposed to Garlon®4 had the opposite occur. It had an increase in Spk value of

0.0009 mm and a decrease in Svk of 0.0012 mm. This shows that there was more surface roughening occurring, possibly due to residue from the herbicide adhering to the material's surface. The control sample, WB1 increased in both Spk and Svk showing an almost equal amount of gain of both surface roughening and pitting. Water can, over time, deteriorate and wear away, although not to the extent of the herbicides, which changed the surface of the brick at a slightly higher rate.

Table 8.2. Changes in Colorimeter Results for the Brick Samples.

Data Name	**L*(C)**	**a*(C)**	**b*(C)**	**E*(C)**
RB1	3.57	-2.34	-3.92	5.8
RB2	0.88	-0.59	-1.82	2.11
RB3	0.02	0.19	-0.63	0.66
GB1	-13.89	3.67	0.14	14.37
GB2	-9.67	6.06	1.54	11.52
GB3	-11.38	0.77	-2.7	11.72
WB1	4.67	0.87	1.66	5.03
WB2	0.14	-0.01	-0.17	0.22
WB3	4.93	0.3	0.12	4.94

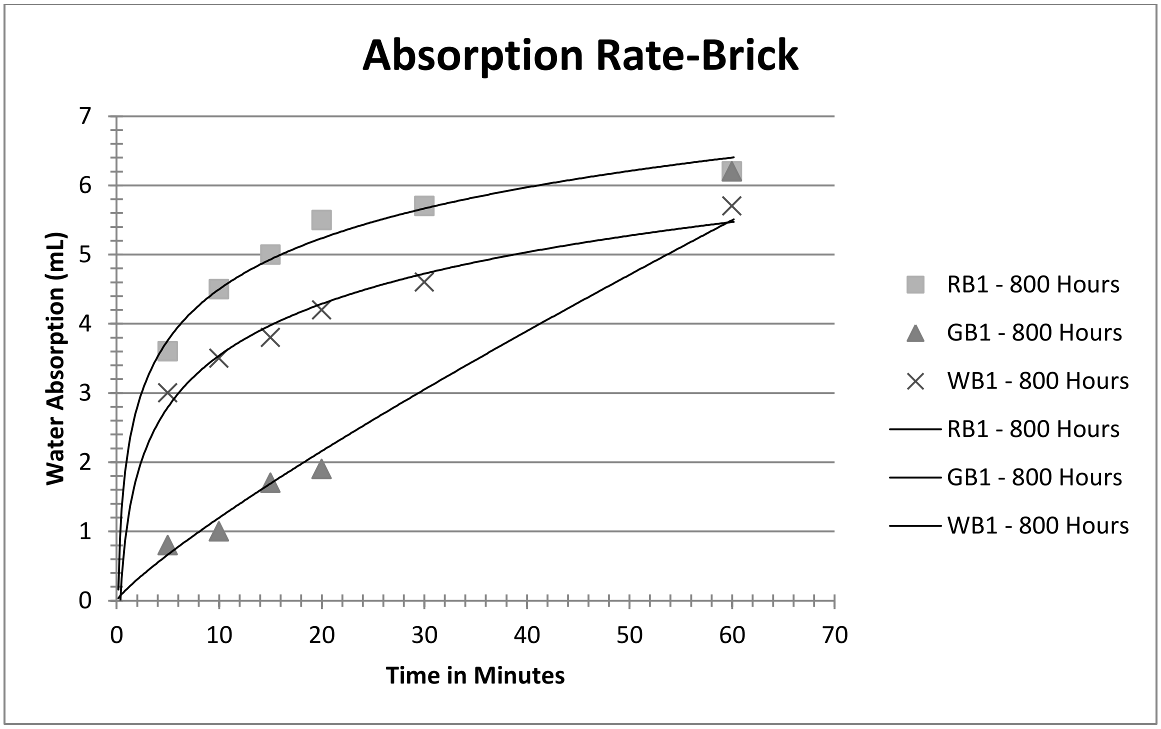
As Table 8.2 shows, the overall color change of the Roundup® brick samples was minimal. All three samples became lighter in color as determined by the increase in L*(C) throughout the experiment (positive numbers indicating a more white/light coloration and negative numbers a more black/dark coloration). However, samples RB1 and RB2 decreased in the "redness" (a*(C)) of their material, but still remained above 16.0 on the redness scale (0.00 is white, negative numbers are green, and positive numbers are red and increase in intensity of red the higher the number, up to 60.00). All samples decreased slightly in "yellowness" (b*(C)) of their material, but still remained high on this scale (0.00 is white, negative numbers are blue, and positive numbers are yellow). The combination of the red and yellow gives the bricks an orange-red coloration. These measurements indicate that the bricks that were treated with Roundup® became lighter in color and a little lighter in yellow/red shades, there was no significant change in color. The samples that were treated with Garlon®4 showed tremendous color change (Appendix B, see photos of Garlon®4 at 0 hours and 800 hours). The lightness of the color decreased significantly. Sample GB1's lightness (L*(C)) decreased by 13.89, quantifying the visible darker color change in the brick. All these samples became darker in appearance. The red/yellow color changes in the materials' color could be due to the orange dye that is incorporated into this herbicide. The dye could stain the surface of the masonry material on contact and over time, through absorption, permanently changing the color of the material. The lightness of the control brick samples varied from an increase of only 0.14 (WB2) to 4.67 (WB1). Some of the color change, lightness/darkness, in these materials is likely caused by dirt being cleansed from the surface of the material through the continuous condensation cycles within the QUV. Otherwise, there was very minimal color change to these samples. The water could have helped "washout" some of the color, especially combined with the UV light.

The pH of all the samples treated with Roundup® increased, becoming less acidic, throughout the experiment. All the samples that were sprayed with Garlon®4 increased in pH level, becoming less acidic. All samples had an initial pH reading of 5 prior to exposure to herbicides. All of them decreased in acidity to a pH of 6 by 337 hours and remained at that pH level for the remainder of the test. All three control samples started with a pH level of 5 and like the other brick samples, increased in pH becoming less acidic, to a pH level of 6. This change in pH is similar to the measurements gathered in all of the brick samples (Roundup® and Garlon®4 samples), which indicate that exposure to Roundup® and Garlon®4 have no measureable effect on brick when compared to brick exposed to deionized water.

The X-ray defraction data showed no significant increases in compositional makeup to the sample by any of the treatments (Roundup®, Garlon®4, or DI water). (Appendix F) The main components in Roundup® are salt compounds that should be visible by the XRD, however, the lack of salt residue in the XRD analysis is likely due to the solubility of the salts and the samples' exposure in the wet environment of the QUV. The main components of Garlon®4 are organic in nature and were not expected to be visible in the XRD analysis.

The FT-IR results showed no evidence of Roundup® herbicide on any of the substrates when their spectra were compared side by side. However, Garlon®4 was found on the brick sample (Figure 7.5). The spectrum for sample GB2 showed the same peaks that were identified as ingredients in the chemical makeup of the herbicide. It was also found that Garlon®4 was creating an iron salt complex on the surface of the brick sample. This suggests that Garlon®4 was still present on the sample and was actually creating something else on the surface of the material.

As shown in Figure 8.1, the absorption rate of the brick exposed to Roundup® was greater than the control brick sample, while the Garlon®4 brick sample had a lower absorption rate. The rate of absorption of water into a masonry material was not constant. Most of the masonry's capacity was filled within the first few minutes of exposure, after which the rate decreases and slows until it remains at a constant level.



RB1 – 800 Hours – $y = 1.0614\ln(x) + 2.0563$, $R^2 = 0.967$

GB1 – 800 Hours - $y = 0.1693x^{0.8501}$, $R^2 = 0.9613$

WB1 – 800 Hours - $y = 1.0761\ln(x) + 1.0643$, $R^2 = 0.9662$

Figure 8.1. Comparison of the Absorption Rates of the Brick Samples.

The rate of absorption equation is a second order kinetics equation. The higher absorption rate of the sample treated with Roundup® could be due to the salt forming on and in the cracks and crevices of the brick. As the salt forms it expands and causes openings to widen and more pitting and cracking to occur. Then, water in the environment can wash out the salt leaving behind larger openings and pores for water to get into the material, which can lead to a faster rate of decay, especially if the brick continues to be exposed to Roundup® herbicide. The Garlon®4 brick sample has a lower rate of absorption than the control sample. This could be due to the hydrophobic makeup of the herbicide that coats the surface of the material and does not seem to wash away.

Limestone

The results for the limestone samples will be examined, compared, and discussed in this section.

Table 8.3. Results of the Physical Tests on the Limestone Samples.

Sample	Herbicide	Salt Formation After 337 Hours QUV	Salt Formation After 800 Hours QUV	Δmass (g)	ΔSk (in mm)	ΔSpk (in mm)	ΔSvk (in mm)
RL1	Roundup®	No	No	0.06	-0.008	0.0015	-0.0014
RL2	Roundup®	No	No	0.07	---	---	---
RL3	Roundup®	No	No	0.07	---	---	---
GL1	Garlon®4	No	No	0.95	0.0061	-0.0005	-0.0065
GL2	Garlon®4	No	No	0.82	---	---	---
GL3	Garlon®4	No	No	0.92	---	---	---
WL1	Control	No	No	-0.01	-0.006	-0.0002	-0.0005
WL2	Control	No	No	0	---	---	---
WL3	Control	No	No	0	---	---	---

As shown in Table 8.3, samples that were sprayed with Roundup® (RL1, RL2, RL3) did not form efflorescence. However, there was a slight translucent haze on the surface of the samples that might have been a thin layer beginning to form, but there were no large “peaks” or areas of significant concentrations of the salt. This could be due to the lack of cracks, large openings, and pores in the limestone, and the coloring of the limestone made it difficult to clearly see salt formation. Samples treated with Garlon®4 did not develop a white haze or signs of salt formation. Control samples treated with deionized water (WL1, WL2, WL3) showed no visible physical changes in appearance nor efflorescence.

All three limestone samples treated with Roundup® increased in mass throughout the experiment. This could be attributed to the efflorescence concentration on and possibly within the masonry material. All limestone samples treated with Garlon®4 increased in weight throughout the experiment. This weight increase could be due to the weight of the herbicide adhering to the limestone. It was remarked that upon spraying that the Garlon®4 herbicide appeared more viscous than the other two solutions. The control samples showed very little, if any, change in weight. Sample WL1 decreased in weight by 0.01 g and neither sample WL2 nor WL3 had any change in weight. This shows no signs of absorption and retention by the material to water during the QUV cycles.

The Spk for sample RL1 increased by 0.0015 mm and the Svk of the sample decreased by 0.0014 mm. This shows almost a balancing between the peaks and valleys. It shows that there is more surface roughening occurring on the limestone sample than pitting. This could be due to the smoothness of the material and the translucent haze that was starting to develop on the surface of the material, which may have been a thin layer of efflorescence starting to develop. This would cause an increase in the surface roughness but no pitting would be occurring because it is still superficial because of the smoothness of the limestone. The Spk for sample GL1 decreased by 0.0005 mm and the Svk also decreased by 0.0065 mm. This suggests that there was filling in of original pits and cracks making the surface smoother, especially in filling in the valleys. The control sample, WL1, also decreased slightly in both the Spk value and the Svk value, showing signs of some wear and natural smoothing of the limestone by water.

Table 8.4. Changes in Colorimeter Results for the Limestone Samples.

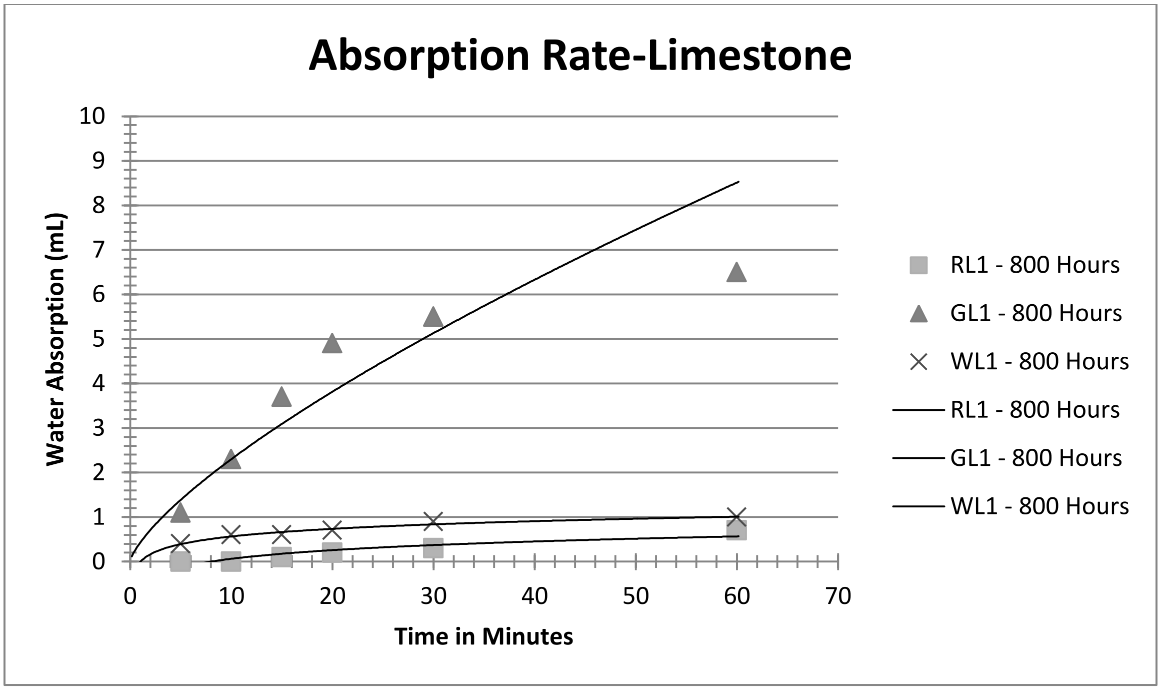
Data Name	L*(C)	a*(C)	b*(C)	E*(C)
RL1	2.07	-0.45	-1.15	2.41
RL2	2.46	-0.54	-1.11	2.75
RL3	3.06	-0.57	-1.29	3.37
GL1	-18.41	1.38	5.69	19.32
GL2	-16.08	0.97	5.06	16.89
GL3	-16.69	1.11	4.89	17.43
WL1	2.55	-0.57	-1.1	2.84
WL2	2.49	-0.46	-0.88	2.68
WL3	2.88	-0.54	-1.12	3.14

As Table 8.4 shows, there was minimal change in the colorimetry measurements for the Roundup® limestone samples. Overall, all samples got lighter in color while decreasing slightly in their levels of "redness" and yellowness." The limestone samples that were exposed to Garlon®4 herbicide showed a significant change in color after being sprayed with the herbicide at 0 hours and 337 hours. These samples decreased significantly in lightness. Sample GL1's ΔL*(C) was -18.41. Sample GL2's ΔL*(C) was -16.08. And sample GL3's ΔL*(C) was -16.69. However, the changes in the samples' a*(C) and b*(C) were not significant, although both increased in all the samples. Overall, sample GL1's color changed +19.32, GL2's changed +16.89, and GL3 had a change of +17.43. These changes in color were likely caused by the orange dye present in the herbicide. There was minimal change in the color of the control samples throughout the experiment. All control samples decreased in “redness” and “yellowness” by less than 1.12. This shows an overall low color change in the control group.

Limestone is naturally basic. The pH level of the Roundup®-limestone samples increased as the masonry materials became more alkaline throughout the experiment. RL1 and RL3 increased from a pH level of 6 to 7, and RL2 increased from a pH level of 5 to 7. All three samples treated with Garlon®4 started with a pH measurement of 6. At 337 hours, only sample GL1 had a change of pH; an increase to a pH of 7. It increased again at 800 hours, with a pH of 8. However, at 800 hours, GL2 and GL3 also increased in pH to 7. All three control samples changed pH from 6 before any treatment to pH 7 after 337 hours in the QUV.

The X-ray defraction data showed no significant increase in compositional make up to the limestone by any of the herbicides tested. This is most likely due to the same reasons given on page 83 in the brick analysis.

There were no traces of Roundup® identified on the limestone samples that were tested using the FT-IR. This could be due to the solubility of the salts and the samples’ exposure to the wet environment in the QUV. However, the peaks for Garlon®4 were present on the spectrum for limestone, suggesting that the herbicide left residue on the samples after treatment.



RL1 – 800 Hours – $y = 0.281\ln(x) – 0.5848$
$R^2 = 0.8462$

GL1 – 800 Hours - $y = 0.4276x^{0.7305}$
$R^2 = 0.8982$

WL1 – 800 Hours - $y = 0.2477\ln(x) - 0.0065$
$R^2 = 0.9545$

Figure 8.2. Absorption Rate Comparison of the Limestone Samples.

Limestone is a harder masonry material, and has small pores. This is reflected in the absorption rates of the samples (Figure 8.2). The rate of absorption of water by the sample exposed to Roundup® was lower than the control's rate of absorption. This could be due to the smooth surface of the material. The salt formed directly on the surface of the limestone sample and because of the smoothness and evenness of the surface, there were no immediate cracks and crevices for the salt to penetrate. Residue from the herbicide might have remained on the surface blocking some of the pores. The limestone sample treated with Garlon®4 is very different and does not reflect a likeness to the control sample. It is likely that there was a hole in the putty during this experiment, which caused the DI water to leak out, thus skewing the results.

Concrete

The results for all the concrete samples will be examined, compared, and discussed in this section.

Table 8.5. Results of the Physical Tests on the Concrete Samples.

Sample	Herbicide	Salt Formation After 337 Hours QUV	Salt Formation After 800 Hours QUV	Δmass	ΔSk (in mm)	ΔSpk (in mm)	ΔSvk (in mm)
RC1	Roundup®	No	No	-0.24	-0.005	-0.134	0.08
RC2	Roundup®	No	Yes	-0.16	---	---	---
RC3	Roundup®	No	Yes	-0.19	---	---	---
GC1	Garlon®4	No	No	0.64	0.001	-0.098	-0.23
GC2	Garlon®4	No	No	0.58	---	---	---
GC3	Garlon®4	No	No	0.5	---	---	---
WC1	Control	No	No	-0.18	0.006	0.026	0.19
WC2	Control	No	No	-0.23	---	---	---
WC3	Control	No	No	-0.22	---	---	---

As shown in Table 8.5, samples treated with Roundup® (RC1, RC2, RC3) developed efflorescence spots on the surface. Small concentrations of efflorescence developed on the rough edges of the surface of the concrete, building up rather than out across the face of the material. Samples sprayed with Garlon®4 did not develop efflorescence on the surface of the material. There was no evidence of efflorescence formation on the surface of the samples treated with deionized water.

The overall mass of the samples exposed to Roundup® decreased. There was an initial decrease in weight during the first 337 hours in the QUV and an increase in weight during the second phase in the QUV. This initial loss of weight followed by the increase could be due to material breaking loose during handling or while in the QUV during the wet/dry cycles. The increase in weight could be due to the efflorescence present on the material after 800 hours in the QUV. All three samples that were treated with Garlon®4 herbicide increased in weight throughout the experiment. This increase weight follows the trend of samples treated with Garlon®4 herbicide increasing in weight, which could be the result of the liquid adhering to or being absorbed and retained by the material. It could be sticking to the surface of the material like a layer of paint, causing the color change and weight increase. All three control samples had an initial decrease in weight, then an increase in weight. The initial weight loss could be due to pieces of the cement sample breaking off during handling or while in the QUV. It could also be caused by the water, soaking the materials that bind the cement together, making them weaker, which would eventually lead to breaking down and further deterioration. The gain of weight could be the absorption of water throughout the experiment.

The Spk of sample RC1 decreased significantly throughout the experiment, becoming less rough and smoother by 0.1324 mm. On the other hand, the Svk of the sample increased by 0.08 mm. This shows that the surface became more pitted and decreased in surface roughness. The smoothing could be from the salt formation on the surface of the material, but the increase of pitting suggests the opening of more pores and cracks in the surface. Both the Spk and the Svk of sample GC1 decreased. The Spk decreased by 0.098 mm suggesting a decrease in surface roughness and a more uniform surface texture. The Svk decreased by 0.23 mm suggesting a reduced number of valleys and no pitting occurring to the surface of the material. Overall, there was a uniform smoothing of the surface of this material suggesting that something was covering or filling in the cracks and pits on the surface of the material. The control sample, WC1 increased in both Spk and Svk values, again suggesting the normal degradation of surface wear by water. Water can erode material and in concrete can aid in loosening aggregate causing chips to fall off or pitting to occur, all of which would increase surface roughness.

Table 8.6. Changes in Colorimeter Results for the Concrete Samples.

Data Name	**L*(C)**	**a*(C)**	**b*(C)**	**E*(C)**
RC1	6.79	-0.15	1.51	6.96
RC2	4.9	0.98	1	5.25
RC3	3.37	0.63	0.07	3.43
GC1	0.91	1.29	5.04	5.28
GC2	-3.83	1.23	4.37	5.94
GC3	-7.45	1.07	1.6	7.69
WC1	5.07	0.18	0.28	5.08
WC2	4.56	0.16	0.75	4.62
WC3	2.71	1.43	1.49	3.41

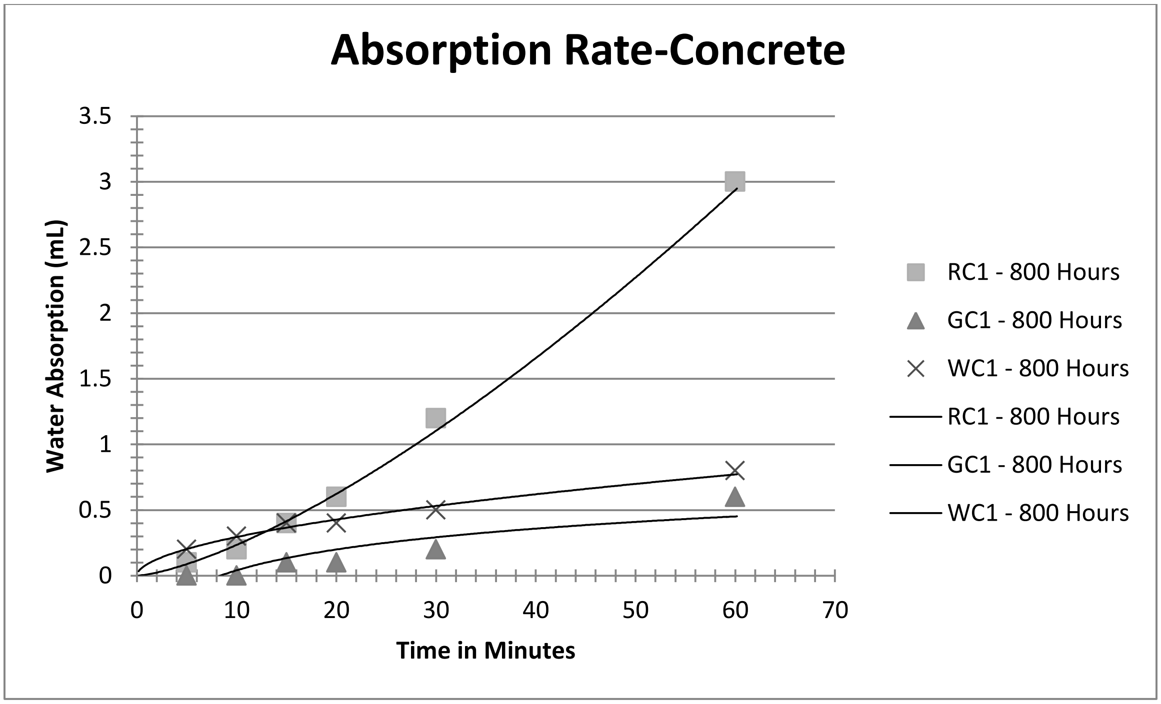
Most of the color change that occurred in the Roundup®-concrete samples was the change in lightness of the material (Figure 8.6). These samples became lighter throughout the experiment. Overall, there was very little change in a*(C) and b*(C). This shows no significant color change due to Roundup® herbicide exposure. There was a change of color in the Garlon®4 samples. Sample GC1 increased in L*(C) by 0.91, but samples GC2 and GC3 decreased in L*(C), becoming darker in color. All the samples increased slightly (maximum of +1.29) in a*(C) and in b*(C) (maximum of +5.04). Overall, the range of color change was +5.28 to +7.69. All of the control samples had increases in L*(C), a*(C), and b*(C). None were significant and shows the possibility of the material's surface being cleaned by the water throughout the experiment and possibly "washed out naturally" by the UV light.

The pH of all the Roundup® samples was initially 8. Samples RC1 and RC2 decreased in pH, dropping to 7 after 337 hours, while sample RC3 did not decrease in pH until after 800 hours. Sample GC1 had an initial pH of 7. However, after 337 hours in the QUV, after the first round of herbicide treatment, the pH of the sample dropped to 6. Samples GC2 and GC3 both had a constant pH of 7 throughout the experiment. All three control samples had a pH of 7 before treatment. All increased to a pH of 8 at 337 hours. Sample WC2 then changed back to a pH of 7 at 800 hours.

The X-ray defraction data showed no significant increase in compositional make up to the limestone by any of the herbicides tested. This is most likely due to the same reasons given on page 83 in the brick analysis.

According to the FT-IR data, there were no traces of Roundup® on the concrete samples. However, there were traces of Garlon®4 on the concrete samples that were exposed to that herbicide. This suggests that Garlon®4 was still present on the sample after treatment.

As shown in Figure 8.3, the concrete sample that was exposed to Roundup® herbicide had a greater rate of absorption than the control sample, while the sample treated with Garlon®4 herbicide had a lower rate of absorption at the end of the experiment. The higher rate of absorption could be due to the expansion of the material's pores by salt formation. While the lower rate of absorption could be due to a blockage of the pores by the hydrophobic Garlon®4 herbicide.



RC1 – 800 Hours – $y = 0.0091x^{1.4116}$ $R^2 = 0.9931$

GC1 – 800 Hours - $y = 0.2292\ln(x) - 0.4869$ $R^2 = 0.7738$

WC1 – 800 Hours - $y = 0.0856x^{0.5367}$ $R^2 = 0.9837$

Figure 8.3. Absorption Rate Comparison of the Concrete Samples.

Granite

The results for the granite samples will be examined, compared, and discussed in this section.

Table 8.7. Results of the Physical Tests on the Granite Samples.

Sample	Herbicide	Salt Formation After 337 Hours QUV	Salt Formation After 800 Hours QUV	Δmass	ΔSk (in mm)	ΔSpk (in mm)	ΔSvk (in mm)
RG1	Roundup®	No	No	0.01	-0.0066	-0.005	-0.0025
RG2	Roundup®	No	No	0.01	---	---	---
RG3	Roundup®	No	No	0.02	---	---	---
GG1	Garlon®4	No	No	0.06	0.0091	0.032	0.096
GG2	Garlon®4	No	No	0.07	---	---	---
GG3	Garlon®4	No	No	0.06	---	---	---
WG1	Control	No	No	-0.01	0.0059	0.0119	0.0137
WG2	Control	No	No	0	---	---	---
WG3	Control	No	No	0	---	---	---

As shown in Table 8.7, granite samples that were treated with Roundup® (RG1, RG2, RG3) did not develop visible physical changes to their surface and there was little or no efflorescence on the surface of the materials. Samples that were exposed to Garlon®4 (GG1, GG2, GG3) did not develop efflorescence on the surface of the material. The granite samples that were treated as the controls, with deionized water, (WG1, WG2, WG3) did not show any visible physical changes to the surfaces of the material, including the absence of efflorescence.

The mass of each of the three samples treated with Roundup® remained pretty constant, with no weight change when measured at 337 hours and a slight increase in weight during the second phase of the experiment. Sample RG1 only increased in weight by a total of 0.01g, sample RG2 increased in weight by 0.01g, and sample RG3 increased in weight by 0.02g. This shows very little change to the weight of each of the samples of granite, and it can be determined that these samples' weight was not affected by Roundup® herbicide and its potential for efflorescence formation. Overall, each of the samples treated with Garlon®4 had a slight increase in weight, which could be due to the layer of Garlon®4 that stained the surface of the material after application. This slight increase in all of the samples could be from the materials' ability to retain moisture either from the condensation cycles in the QUV (water) or, as sample GG2 showed, Garlon®4's ability to adhere to the surface of the material. There were no significant changes in weight to any of the control samples. This is expected due to the hardness and low porosity of the material. Granite does not readily soak up water or other solutions that it comes into contact with and therefore does not retain them causing weight gain. Also, the lack of liquid entering the material limits the amount of salt intake as well, decreasing the chance of efflorescence development.

The Spk for the sample RG1 decreased by 0.005 mm and the Svk decreased by 0.0025 mm. This shows a smoothing of the surface, the number of peaks and valleys has decreased since the start of the experiment. The Spk of sample GG1 increased by 0.032 mm and the Svk also increased by 0.096 mm. This increase in both surface roughness and pitting could be attributed to the Garlon®4 herbicide adhering to the surface of the material and (because granite is incredibly smooth to start with) the liquid could have dried unevenly to the surface. The control sample, WG1, also increased in both Spk and

Svk value but not as greatly as the GG1 sample. This could be the result of the usual degradation caused by water on the surface of the granite material.

Table 8.8. Changes in Colorimeter Results for the Granite Samples.

Data Name	**L*(C)**	**a*(C)**	**b*(C)**	**E*(C)**
RG1	1.86	-0.12	0.62	1.96
RG2	0.27	-0.26	-0.44	0.58
RG3	-0.72	-0.2	-0.75	1.06
GG1	-14.53	0.61	13.81	20.06
GG2	-15.67	0.89	9.03	18.11
GG3	-14.5	0.68	13.06	19.53
WG1	-0.08	-0.16	-0.19	0.26
WG2	-1.12	-0.2	0.22	1.16
WG3	0.12	0.04	0.05	0.14

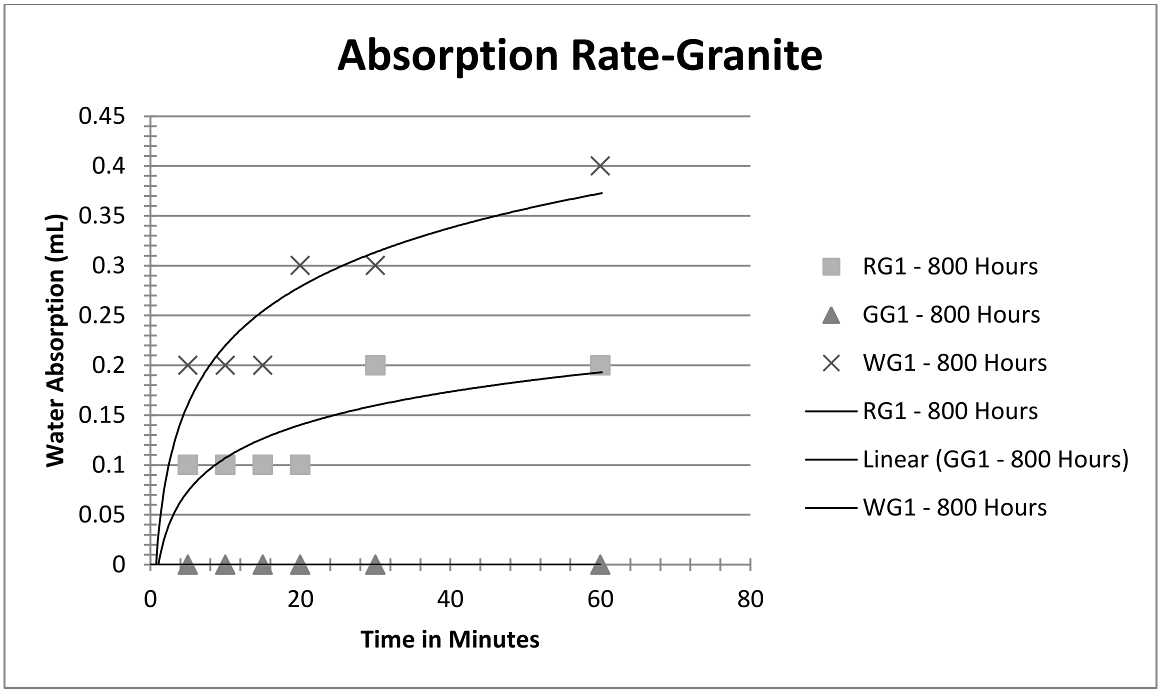
As shown in Table 8.8, there was very minimal color change to any of the Roundup® samples. Sample RG1 had an overall color change of +1.96. It had a ΔL*(C) of +1.86, a Δa*(C) of -0.12, and a Δb*(C) of +0.62. Sample RG2 had an overall color change of +0.58, with a ΔL*(C) of +0.27, a Δa*(C) of -0.26, and a Δb*(C) of -0.44. Sample RG3 had an overall change of +1.06. It had a ΔL*(C) of -0.72, a Δa*(C) of -0.20, and a Δb*(C) of -0.75. All of them decreased in a*(C). All three Garlon®4 treated samples changed in color, becoming darker as the herbicide stained the surface of the materials after each application. These samples had the most significant changes in color. The overall color changes (E*(C)) of the samples were: +20.06, +18.11, and +19.53. All of the samples decreased in L*(C) by at least 14.50. There was minimal change in a*(C). This quantifies the dramatic visible change in these samples as they became darker in color after exposure to Garlon®4. There were no significant changes in the color of the material of the DI water samples.

Herbicide on granite had very little effect on the pH. This lack of change in pH over the course of this experiment is likely due to the lack of chemical change in the granite upon contact with the herbicide. Samples GG1 and GG2 had an original pH of 7 and sample GG3 had a pH of 6. Sample GG1 remained at a constant pH of 7 throughout the experiment. Sample GG2 decreased to a pH of 6 after the first 337 hours in the QUV, but increased back to a pH of 7 after 800 hours in the QUV. Sample GG3 remained at a pH level of 6 for the first 337 hours of the experiment, but increased, becoming pH 7 after 800 hours. The pH of the control samples remained constant, close to 7. These pH levels are consistent with deionized water results.

The X-ray defraction data showed no significant increase in compositional make up to the limestone by any of the herbicides tested. This is most likely due to the same reasons given on page 83 in the brick analysis.

As shown in Figure 7.6, there were no traces of Roundup® on the granite sample that was exposed to that herbicide. This was likely because of the solubility of the salts, the lack of porosity of the granite, and the wet environment the samples encountered in the QUV. However, there was also no evidence of Garlon®4 on any of the granite samples, which is not what was expected after all the evidence suggesting that residue may be left on the material.

Out of all four masonry materials used in this experiment, granite is the hardest with the smallest pore size (Figure 8.4). The control sample had the highest rate of absorption which could mean that both Roundup® and Garlon®4 could have been present on the materials' surface, causing the pores to be blocked and prohibiting water from penetrating the surface.



RG1 – 800 Hours – $y = 0.048\ln(x) - 0.0036$, $R^2 = 0.6451$

GG1 – 800 Hours - $y = 0$, R^2 = N/A

WG1 – 800 Hours - $y = 0.0852\ln(x) + 0.0238$, $R^2 = 0.8118$

Figure 8.4. Absorption Rate of the Granite Samples.

Again, Roundup® salt could have formed on the surface of the material washed off when it came in contact with water.

<u>Possible Errors in the Experimentation</u>

Because of the complexity of this experiment, errors could have occurred during the experiment. Possible errors have been observed and found to be minimal.

First, the experimental design called for the experiment to be completed in 800 hours, which it was. However, the samples were supposed to be sprayed with their designated solution every 200 hours. This did not occur due to QUV pump failure and family emergencies that prevented the machine from being stopped and completing that portion of the experiment on schedule. The samples were treated and pulled out of the

QUV earlier or later than scheduled. However, this was taken into consideration by the experimenter and NCPTT staff, all of whom felt that the new times noted and the experiment should continue. In an uncontrolled environment, the herbicide could be sprayed at random times and not necessarily on a controlled schedule. In the end, the samples were in the QUV for exactly 800 hours and had been treated with their solutions a total of 4 times as originally planned.

Another possible error to consider in this experiment is that the samples were sharing QUV space with samples from another NCPTT project, 5 SO_2 samples. The samples did not appear to have any chemical or physical effect on each other, but for future studies, to reduce possible interferences, the samples should be kept in separate QUVs for the duration of the experiments.

Another difficulty encountered in this experiment is the use of litmus paper to test the pH of the samples. Due to the nature of the samples, it was impossible to liquefy them in order to test the pH using a pH meter, which would have been more accurate. Instead, litmus paper was used because of the solid surfaces of the masonry material and the minimal effects it might have on the samples when it was placed on their surface and sprayed with DI water. However, it was found that the pH readings were all relatively the same, near or around the reading of 7, neutral. It is believed that this is in large part due to the DI water used to adhere the litmus paper to the sample in order to get a reading; the litmus paper could have been reading the pH of the DI water rather than that of the sample. In future research, litmus paper should be replaced by a more accurate way to determine the pH of the sample.

Due to the nature of masonry material and its tendency to absorb DI water initially at a faster rate (within the first couple of minutes of exposure), the absorption test should have been redone and completed while monitoring the water absorption in the first 10 minutes of the experiment. This would ensure more data points available within the time frame when the most water is absorbed. By having more of these points a more accurate rate of absorption could have been measured.

CHAPTER 9

CONCLUSION

Herbicides are used all over the world to rid places of unwanted vegetation. They are used in open fields, home gardens, and landscapes surrounding modern and historic structures, among many other locations. The biological and chemical effects of herbicides on animal health, environmental health, and different vegetation have been studied extensively. However, the physical and chemical effects of herbicides on structural materials, such as stone and masonry, have only been minimally studied.

Using the data generated from this experiment it is concluded that Roundup® herbicide and Garlon®4 herbicide have negative effects on stone and masonry materials.

Roundup® is a salt based herbicide that produced efflorescence on the surface and in the pores and cracks of the stone and masonry materials. When this salt forms after exposure to Roundup® and weather, it expands on and in the materials, thereby widening crevices, cracks, and pores on and within the surface. The salt can be washed away by water/rain but not always before damage is done. The larger openings provide enlarged spaces for water, dirt, and vegetation to get into and further deteriorate the material, which could eventually, over time, lead to structural failure. Nevertheless, this process is not likely to be rapid. It could take several years or even decades for the stone or masonry to significantly weaken if only sprayed with Roundup® once or twice a year. It is a negative process that is causing long-term negative impacts to the material and will eventually lead to degradation. There were no chemical changes observed from the materials' exposure to Roundup®, which means that most of the observable/measurable changes that occurred were physical in nature, such as formation of salt, change in weight (possibly weight gain due to salt accumulation or weight loss due to salt-induced expansion and fragmentation), and increased rate of absorption (increased pore size).

Garlon®4 has a much more immediate observable impact on the stone and masonry materials. As seen in the pictures (Appendix B) and colorimeter data (Appendix D) accumulated throughout the experiment, Garlon®4 stains the surface of the materials, making them darker in appearance. This is caused by the orange dye that the manufacturer puts into the solution to help the human herbicide applicator track where he or she has previously sprayed the solution. The darker appearance of the material changes the appearance and character of the material and the site, and the stone appears less aesthetically pleasing. It was found that the Garlon®4 adheres to the materials' surface, blocking pores and repelling water. Although Garlon®4 may reduce decay through absorption into the material, it causes negative impacts by coating and staining the surface of the material. Garlon®4 residue was detected on some of the materials after 800 hours in the QUV. This shows that some of the herbicide was adhering to the surface. There were no experimental results that suggest that Garlon®4 exposure to the masonry and stone materials caused chemical changes.

For both Garlon®4 and Roundup® herbicides tested in this experiment, it is concluded that they caused physical changes to the brick, limestone, concrete, and granite. These negative effects were mostly physical changes that affected the aesthetics of their surfaces, and, long-term, could affect the structural integrity.

It should be kept in mind that these results should be used as an awareness tool and that these results are not the standard for all masonry materials. This test shows that herbicide can have a negative effect on masonry materials and that users should be aware that these effects exist.

Recommendations for Future Research

There are several avenues a future researcher might take in the further examination of the effects of herbicide on stone and masonry material. Here are a few recommendations:

- Do an abrasion test to measure the hardness of each of the samples of masonry. This test would show any changes in the materials physical structure over time, if any, after being exposed to herbicide. The acid in the herbicide might weaken the structural bonds of the masonry material which could cause the structure to weaken.
- Perform the experiment in a natural environment, outdoors, not in the QUV, which is a weather simulator. The test could be monitored in multiple regions/climates where weather patterns are different and different vegetation might require different herbicide application rates. Increased application rates would mean the herbicide may be applied more times and/or in greater quantities.
- A similar experiment could be used to test the effects of the herbicides on different types of mortar. The materials used to bind the masonry materials together may different susceptibilities to herbicides than the masonry and stone. The mortars have different porosities and chemical structures than the

masonry and stone. Degradation of the mortars may have greater negative effects on structural integrity than effects on stone and masonry.

- Different herbicides could also be tested. Or the same ones, Roundup® and Garlon®4 could be used but in different concentrations.

There are many variations of this test that could be performed to further determine if herbicides affect stone and masonry structures, and many tests that can be performed that will help in determining any chemical or physical effects.

Overall, a less damaging method of unwanted vegetation removal is by hand. For this method, there are no chemicals being introduced onto the historic stone or masonry materials or into the environment. However, this way is not always the most practical due to restrictions on time, labor, and budget. Herbicides such as Roundup® and Garlon®4 are inexpensive means for quickly killing unwanted vegetation. Decision makers (gardeners, landscape managers) should keep in mind that when using these chemicals near historic features that the exposure to herbicides are likely to lead to an increased rate of deterioration of the historic features.

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APPENDIX A

THE USE OF HERBICIDES NEAR MASONRY, NATIONAL PARK SERVICE

SURVEY, JUNE 21, 2010

Herbicides are utilized across the country as effective pest management tools. However, little is known about the impact that they may have on structural materials. The data you provide will help aid in a study conducted by the National Center for Preservation Technology and Training to determine the effects of herbicides on stone and masonry so we can better preserve our nation's historic features. This is solely a masonry study. Thank you for your time.

Site Name:

Location:

Is there an Integrated Pest Management system in place for controlling unwanted vegetation in these areas? ___Yes ___No

Are herbicides used as a form of removing unwanted vegetation (grass, ivy, invasive species, etc)? ___Yes ___No

If yes, please continue the survey, providing information on up to 3 important historic features which herbicide is used on or near to control vegetation.

If not, what technique is being applied to the area to remove unwanted vegetation?

___Mechanical removal
___Hand removal
___ Other ________________________________
___No treatment

Historic Feature #1

1. What is the feature?
   ___House
   ___Fort
   ___Monument
   ___Cemetery
   ___Other________________________________

2. What type(s) of material is the historic feature made of? Check all that apply.
   ___ Brick
   ___ Concrete Block
   ____Natural stone
   ___ Sandstone
   ___ Limestone
   ___ Marble
   ___ Granite
   ___Other ________________________________

3. What is the age of the feature?

4. How would you rank the feature material's current condition?
   ___Poor (badly deteriorated, many cracks, discoloration, and/or efflorescence)
   ___Good (some cracking, discoloration and/or efflorescence)
   ___Excellent (no cracks, no visible signs of deterioration)

5. What type of herbicide is used on the area?
   ___Roundup® (Glyphosate)
   ___Vanquish® (Dicamba)
   ___Garlon®4 (Triclopyyr)
   ___Arsenal® (Imazapyr)
   ___Other________________

6. Is the manufacturer's recommended concentration used? If not, what concentration is applied?
   ___Yes ___No, ___________concentration is applied.

7. How often is the herbicide applied to vegetation?
   ___Daily
   ___Weekly
   ___Monthly
   ___Twice a year
   ___Annually
   ___As needed
   ___Other ______________________________________

8. What month(s) do you typically apply the herbicide?

9. How is the herbicide applied?
   ___ Broad-spray
   ___Cut Surface Spot Treatment (hack and squirt, cut stump, frill)
   ___Stem Injection
   ___Other______________________________________

10. Is it applied directly on the historic structure material?
    ___Yes ___No

11. Are herbicides used in combination with any other chemical compounds such as wetting agents (i.e. Induce®)? If so, please list all combinations and the concentrations of each that are used.
    ___Yes ___ No

12. Has there been a noticeable change in the masonry material since the use of herbicide on/around the feature? If so, please describe it below.

    ___Yes ___ No

13. If you have any additional comments please provide them below.

Repeat for Historic structures #2 and #3

APPENDIX B

SAMPLE PHOTOGRAPHS OVER TIME

ID *0 Hours* *337 Hours* *800 Hours*

RB1

RB2

RB3

RL1

RL2

ID *0 Hours* *337 Hours* *800 Hours*

RL3

RC1

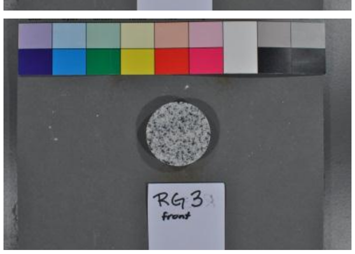
RC2

RC3

RG1

RG2

RG3



ID	0 Hours	337 Hours	800 Hours

GB1

GB2

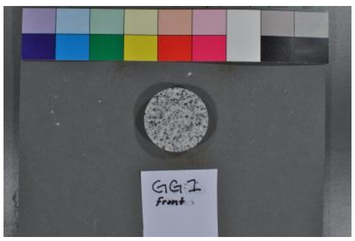
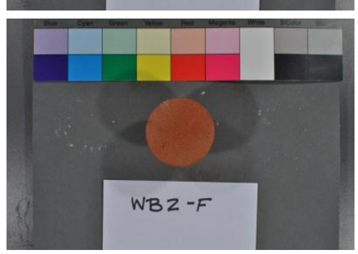
GB3

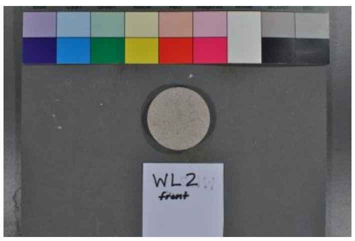
GL1

GL2

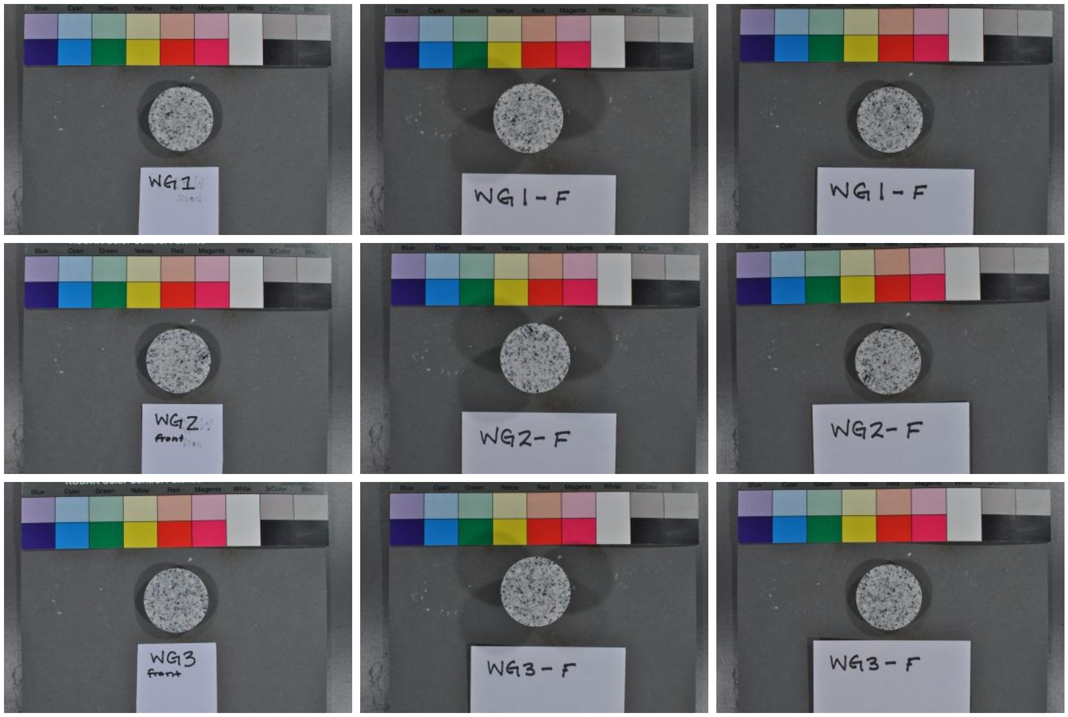
GL3

GC1

ID	*0 Hours*	*337 Hours*	*800 Hours*
GC2			
GC3			
GG1			
GG2			
GG3			
WB1			
WB2			

ID	*0 Hours*	*337 Hours*	*800 Hours*
WB3			
WL1			
WL2			
WL3			
WC1			
WC2			
WC3			

ID *0 Hours* *337 Hours* *800 Hours*



WG1

WG2

WG3

APPENDIX C

FINAL SAMPLE VISUAL SURVEY RESULTS

Survey #	RB1	RB2	RB3	GB1	GB2	GB3	WB1	WB2	WB3
1	3	2	3	5	5	4	5	3	4
2	2	3	2	5	5	3	2	1	1
3	3	0	3	4	4	3	4	4	4
4	3	3	2	4	3	3	3	1	4
5	3	1	3	4	4	3	5	4	4
6	2	2	2	3	2	2	3	2	4
7	2	2	1	4	3	3	1	2	2
8	3	1	2	4	4	3	1	0	0
9	3	1	1	3	3	4	2	1	1
10	4	3	2	5	5	4	0	0	1
11	3	2	2	3	3	3	2	1	3
12	4	2	2	2	2	2	1	1	1
13	2	1	2	3	3	3	1	0	0
14	2	3	2	4	4	2	1	1	1
15	3	1	1	4	4	1	0	0	0
16	3	2	2	4	4	3	1	1	1
17	3	2	2	4	4	2	1	1	1
18	2	2	2	3	2	2	1	0	0
19	3	2	2	4	4	3	3	1	
20	2	2	1	3	3	2	0	0	1
21	4	5	3	4	3	2			
22	3	2	3	4	4	3	1	1	1
23	3	2	2	5	5	3	2	1	1
24	4	3	4	4	4	4	3	1	2
25	2	1	1	5	4	3	0	0	0
26	2	2	1	1	2	2	2	0	2
Average	2.8	2.0	2.0	3.8	3.6	2.8	1.8	1.1	1.6
	RB1	**RB2**	**RB3**	**GB1**	**GB2**	**GB3**	**WB1**	**WB2**	**WB3**
Discoloration	21	15	14	26	26	26	11	5	9
Efflorescence	26	19	23				14	5	7
pits/holes		13		2	1	7		1	2
other		1		2	2				
Comments		cracks		the white color disappeared	smoother surface texture				

Survey #	RL1	RL2	RL3	GL1	GL2	GL3	WL1	WL2	WL3
1	1	1	1	2	2	2	1	1	1
2	1	1	1	2	2	2	1	1	1
3	0	0	0	4	4	4	1	1	1
4	2	0	0	2	3	3	0	1	0
5	1	1	2	4	4	4	3	3	2
6	1	1	1	2	2	2	1	1	1
7	1	1	1	3	3	3	2	1	1
8	0	0	0	3	3	3	0	0	0
9	1	1	1	4	4	3	1	1	1
10	1	1	1	5	5	5	0	1	0
11	1	1	1	2	2	2	2	2	1
12	1	1	1	2	2	2	1	1	1
13	0	0	0	1	1	1	0	0	0
14	1	1	1	2	3	3	1	1	1
15	0	0	0	1	2	2	0	0	0
16	1	1	1	4	4	4	2	1	1
17	1	1	1	3	3	3	1	1	1
18	0	0	0	2	2	2	1	0	0
19	0	1	1	5	4	3	1	1	1
20	0	1	1	3	3	3	1	0	0
21	0	0	0	4	4	4	0	0	0
22	1	2	1	3	3	3	2	2	1
23	1	2	2	3	3	3	2	2	1
24	1	1	2	4	4	4	1	2	2
25	0	1	0	3	4	5	1	1	0
26		0	0	2		2	0	0	0
Average	0.7	0.8	0.8	2.9	3.0	3.0	1.0	1.0	0.7
	RL1	**RL2**	**RL3**	**GL1**	**GL2**	**GL3**	**WL1**	**WL2**	**WL3**
Discoloration	2	6	5	26	26	26	11	7	3
Efflorescence		1							
pits/holes	1			1	3	2	1	3	
other									
Comments									

Survey #	RC1	RC2	RC3	GC1	GC2	GC3	WC1	WC2	WC3
1	1	1	1	2	2	2	1	1	1
2	2	2	2	3	3	3	1	2	2
3	0	0	0	2	2	2	0	0	0
4	1	0	0	2	3	2	1	1	1
5	2	2	2	4	4	4	2	2	1
6	1	1	1	3	3		2	2	1
7	2	3	1	4	4	4	2	1	1
8	1	0	0	3	3	3	0	0	0
9	2	2	1	3	3	4	1	0	0
10	4	0	0	3	4	4	1	0	1
11	2	1	2	3	2	2	1	1	2
12	2	2	2	3	3	3	1	1	1
13	1	0	1	1	2	2	0	0	0
14	2	2	3	3		3	2	1	1
15	0	0		0	1	1	0	0	0
16	2	2	2	4	4	4	1	1	1
17	1	1	1	2	2	2	1	1	1
18	0	0	0	2	2	2	1	0	0
19	1	1	1	3	3	3	1	1	1
20	0	0	0	2	2	2	0	0	0
21	0	0	2	3	3	3	3	2	1
22	2	2	1	2	3	2	2	1	1
23	2	1	1	3	3	3	1	1	1
24	1	1	1	4	4	4	3	2	2
25	0	0	1	2	3	4	0	0	0
26	0	0	0	3	2	2	2	0	0
Average	1.2	0.9	1.0	2.7	2.8	2.8	1.2	0.8	0.8
	RC1	**RC2**	**RC3**	**GC1**	**GC2**	**GC3**	**WC1**	**WC2**	**WC3**
Discoloration	11	8	9	24	24	25	10	5	6
Efflorescence	3		3	1	2	1	1		1
pits/holes	2			1					
other	1							1	
Comments	missing stones							materials loss	

Survey #	RG1	RG2	RG3	GG1	GG2	GG3	WG1	WG2	WG3
1	1	1	1	3	5	4	1	1	1
2	1	1	1	5	5	5	2	1	2
3	0	0	0	4	5	4	0	0	0
4	0	1	1	3	4	3	0	0	1
5	1	4	3	5	5	5	3	2	2
6	1	1	1	5	5	5	1	1	1
7	1	1	1	4	5	4	1	1	2
8	2	2	0	4	4	4	2	2	0
9	0	0	0	5	5	5	0	0	2
10	2	2	1	5	5	5	3	1	3
11	1	1	1	2	3	3	1	1	1
12	2	2	1	5	5	5	2	2	1
13	0	0	0	3	3	3	0	0	0
14	1	1	1	4	3	3	1	1	1
15	0	0	0	3	3	3	0	0	0
16	1	1	1	4	4	4	1	1	1
17	1	1	1	4	4	4	1	1	3
18	1	1	0	3	2	2	1	1	1
19	1	1	1	2	3	3	1	1	1
20	1	1	0	3	4	4	1	0	0
21	2	2	2	4	4	4	2	1	2
22	2	2	1	4	4	3	3		3
23	1	1	1	4	4	4	2	1	1
24	1	1	1	4	4	4	2	2	1
25	0	0	0	3	4	4	1	0	0
26	1	0	0	5	5	3	0	0	0
Average	1.0	1.1	0.8	3.8	4.1	3.8	1.2	0.8	1.2
	RG1	**RG2**	**RG3**	**GG1**	**GG2**	**GG3**	**WG1**	**WG2**	**WG3**
Discoloration	8	7	2	26	26	26	10	6	9
Efflorescence				4	23	20	1		1
pits/holes				2	3	2	1		
other	2	2	2		1		1	1	2
Comments	more black spots	more black spots	more spots; lighter materials seem to be fading		scratches		more spots	lighter materials seem to be fading	more spots; lighter materials seem to be fading

APPENDIX D

COLORIMETER DATA

Colorimetry measurements of each sample before treatment (0 hours).

	Data Name	**L*(C)**	**a*(C)**	**b*(C)**
1	RB1 (12/14/2010 1:51:21 PM)	44.49	22.47	32.26
2	RB2 (12/14/2010 1:52:02 PM)	49.8	16.93	23.63
3	RB3 (12/14/2010 1:52:26 PM)	51.27	21.84	34.25
4	RL1 (12/14/2010 1:56:06 PM)	69.52	2.39	9.97
5	RL2 (12/14/2010 1:56:34 PM)	71.44	2.27	9.46
6	RL3 (12/14/2010 1:57:06 PM)	71.69	2.18	9.63
7	RC1 (12/14/2010 2:00:38 PM)	49.76	5.62	17.72
8	RC2 (12/14/2010 2:01:13 PM)	55.17	2.37	16.17
9	RC3 (12/14/2010 2:01:39 PM)	55.21	3.08	17.03
10	RG1 (12/14/2010 2:04:43 PM)	73.03	-0.28	0.21
11	RG2 (12/14/2010 2:05:08 PM)	66.93	-0.56	1.7
12	RG3 (12/14/2010 2:05:33 PM)	71.08	-0.54	2.15
13	GB1 (12/14/2010 1:53:00 PM)	50.98	13.17	21.33
14	GB2 (12/14/2010 1:53:32 PM)	50.62	13.24	25.21
15	GB3 (12/14/2010 1:54:05 PM)	49.4	18.26	29.51
16	GL1 (12/14/2010 1:57:37 PM)	71.33	2.32	9.87
17	GL2 (12/14/2010 1:58:21 PM)	71.56	2.34	9.91
18	GL3 (12/14/2010 1:58:46 PM)	72.17	2.27	10.27
19	GC1 (12/14/2010 2:02:03 PM)	48.78	4.72	16.52
20	GC2 (12/14/2010 2:02:27 PM)	57.47	3.24	15.2
21	GC3 (12/14/2010 2:02:55 PM)	49.36	6.13	18.28
22	GG1 (12/14/2010 2:05:56 PM)	70.49	-0.45	1.14
23	GG2 (12/14/2010 2:06:21 PM)	73.5	-0.39	0.28
24	GG3 (12/14/2010 2:06:45 PM)	71.98	-0.37	1.68
25	WB1 (12/14/2010 1:54:36 PM)	42.53	14.05	19.98
26	WB2 (12/14/2010 1:55:04 PM)	45.85	21.39	27.05
27	WB3 (12/14/2010 1:55:29 PM)	46.44	11.81	23.58
28	WL1 (12/14/2010 1:59:11 PM)	71.06	2.31	10.23
29	WL2 (12/14/2010 1:59:39 PM)	70.51	2.34	10.13
30	WL3 (12/14/2010 2:00:05 PM)	72.11	2.31	10.11
31	WC1 (12/14/2010 2:03:21 PM)	54.47	3.45	17.62
32	WC2 (12/14/2010 2:03:48 PM)	56.6	3.1	16.51
33	WC3 (12/14/2010 2:04:15 PM)	52.09	2.08	14.61
34	WG1 (12/14/2010 2:07:16 PM)	72.72	-0.3	0.45
35	WG2 (12/14/2010 2:07:40 PM)	73	-0.45	0.85
36	WG3 (12/14/2010 2:08:08 PM)	70.73	-0.5	0.08

Colorimetry measurements recorded after 337 hours in the QUV.

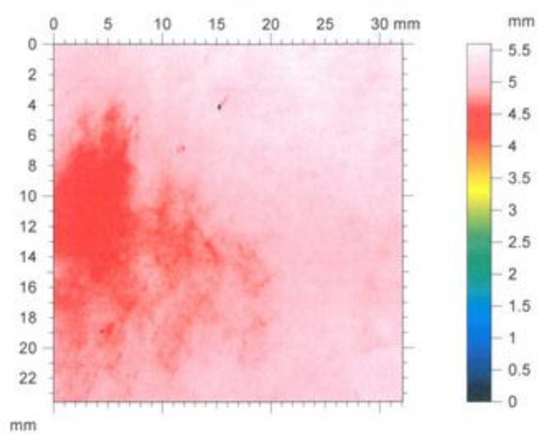
	Data Name	**L*(C)**	**a*(C)**	**b*(C)**
1	RB1 (1/7/2011 3:51:08 PM)	46.68	21.04	30.57
2	RB2 (1/7/2011 10:33:01 AM)	51.19	15.81	20.77
3	RB3 (1/7/2011 10:33:45 AM)	50.94	22.28	34.6
4	RL1 (1/7/2011 10:37:22 AM)	70.81	2.03	9.38
5	RL2 (1/7/2011 10:38:17 AM)	73.55	1.76	8.31
6	RL3 (1/7/2011 10:38:57 AM)	73.14	1.8	8.76
7	RC1 (1/7/2011 10:43:16 AM)	57.43	4.13	15.71
8	RC2 (1/7/2011 10:43:55 AM)	59.9	3.15	16.28
9	RC3 (1/7/2011 10:44:30 AM)	60.99	3.18	15.98
10	RG1 (1/7/2011 4:36:12 PM)	75.98	-0.38	0.43
11	RG2 (1/7/2011 10:49:13 AM)	67.74	-0.76	1.14
12	RG3 (1/7/2011 10:49:46 AM)	71.51	-0.62	1.51
13	GB1 (1/7/2011 3:50:01 PM)	39.1	16.71	22.52
14	GB2 (1/7/2011 10:34:34 AM)	43.92	18.48	27.74
15	GB3 (1/7/2011 10:35:20 AM)	41.98	19.51	29.55
16	GL1 (1/7/2011 10:39:37 AM)	55.69	3.67	16.23
17	GL2 (1/7/2011 10:40:15 AM)	55.95	3.58	16.82
18	GL3 (1/7/2011 10:40:50 AM)	57.19	3.73	17.98
19	GC1 (1/7/2011 10:45:07 AM)	45.6	5.18	20.04
20	GC2 (1/7/2011 10:45:45 AM)	42.44	5.41	20.09
21	GC3 (1/7/2011 10:46:28 AM)	44.3	6.38	19.05
22	GG1 (1/7/2011 10:56:44 AM)	64.74	-0.96	10.7
23	GG2 (1/7/2011 10:50:28 AM)	44	-0.35	11.51
24	GG3 (1/7/2011 10:51:05 AM)	52.97	0.22	12.26
25	WB1 (1/7/2011 3:50:33 PM)	46.63	15.01	21.92
26	WB2 (1/7/2011 10:36:05 AM)	45.82	21.49	27.05
27	WB3 (1/7/2011 10:36:28 AM)	51.1	11.4	22.85
28	WL1 (1/7/2011 10:41:26 AM)	73.37	1.79	9.05
29	WL2 (1/7/2011 10:42:00 AM)	71.89	1.94	9.31
30	WL3 (1/7/2011 10:42:35 AM)	74.29	1.78	9.02
31	WC1 (1/7/2011 10:47:06 AM)	60.26	3.33	17.52
32	WC2 (1/7/2011 10:47:38 AM)	58.75	3.72	17.78
33	WC3 (1/7/2011 10:48:11 AM)	54.28	3.71	15.65
34	WG1 (1/7/2011 10:51:42 AM)	72.77	-0.39	0.2
35	WG2 (1/7/2011 10:52:26 AM)	72.03	-0.67	0.7
36	WG3 (1/7/2011 10:52:59 AM)	70.64	-0.64	0.4

Colorimetry measurements of the samples at the completion of the experiment.

	Data Name	L*(C)	a*(C)	b*(C)
1	RB1 (3/15/2011 11:43:39 AM)	48.06	20.13	28.34
2	RB2 (3/15/2011 11:34:37 AM)	50.68	16.34	21.81
3	RB3 (3/15/2011 11:29:54 AM)	51.29	22.03	33.62
4	RL1 (3/15/2011 11:35:45 AM)	71.59	1.94	8.82
5	RL2 (3/15/2011 11:37:49 AM)	73.9	1.73	8.35
6	RL3 (3/15/2011 11:42:21 AM)	74.75	1.61	8.34
7	RC1 (3/16/2011 9:22:05 AM)	56.55	5.47	19.23
8	RC2 (3/15/2011 11:38:16 AM)	60.07	3.35	17.17
9	RC3 (3/15/2011 11:28:35 AM)	58.58	3.71	17.1
10	RG1 (3/15/2011 11:40:14 AM)	74.89	-0.4	0.83
11	RG2 (3/15/2011 11:39:09 AM)	67.2	-0.82	1.26
12	RG3 (3/15/2011 11:35:20 AM)	70.36	-0.74	1.4
13	GB1 (3/15/2011 11:41:00 AM)	37.09	16.84	21.47
14	GB2 (3/15/2011 11:39:34 AM)	40.95	19.3	26.75
15	GB3 (3/15/2011 11:33:49 AM)	38.02	19.03	26.81
16	GL1 (3/15/2011 11:27:57 AM)	52.92	3.7	15.56
17	GL2 (3/15/2011 11:36:58 AM)	55.48	3.31	14.97
18	GL3 (3/15/2011 11:27:20 AM)	55.48	3.38	15.16
19	GC1 (3/16/2011 9:23:54 AM)	49.69	6.01	21.56
20	GC2 (3/15/2011 11:32:30 AM)	53.64	4.47	19.57
21	GC3 (3/15/2011 11:36:21 AM)	41.91	7.2	19.88
22	GG1 (3/16/2011 9:21:36 AM)	55.96	0.16	14.95
23	GG2 (3/15/2011 11:30:32 AM)	57.83	0.5	9.31
24	GG3 (3/15/2011 11:31:24 AM)	57.48	0.31	14.74
25	WB1 (3/15/2011 11:26:17 AM)	47.2	14.92	21.64
26	WB2 (3/15/2011 11:21:55 AM)	45.99	21.38	26.88
27	WB3 (3/15/2011 11:43:00 AM)	51.37	12.11	23.7
28	WL1 (3/15/2011 11:32:05 AM)	73.61	1.74	9.13
29	WL2 (3/15/2011 11:25:21 AM)	73	1.88	9.25
30	WL3 (3/15/2011 11:26:44 AM)	74.99	1.77	8.99
31	WC1 (3/16/2011 9:19:36 AM)	59.54	3.63	17.9
32	WC2 (3/15/2011 11:24:42 AM)	61.16	3.26	17.26
33	WC3 (3/15/2011 11:41:40 AM)	54.8	3.51	16.1
34	WG1 (3/15/2011 11:29:17 AM)	72.64	-0.46	0.26
35	WG2 (3/15/2011 11:24:14 AM)	71.88	-0.65	1.07
36	WG3 (3/15/2011 11:33:25 AM)	70.85	-0.46	0.13

APPENDIX E

LASER PROFILOMETER DATA



Parameters calculated on the surface RB1 12-7-2010

Amplitude Parameters

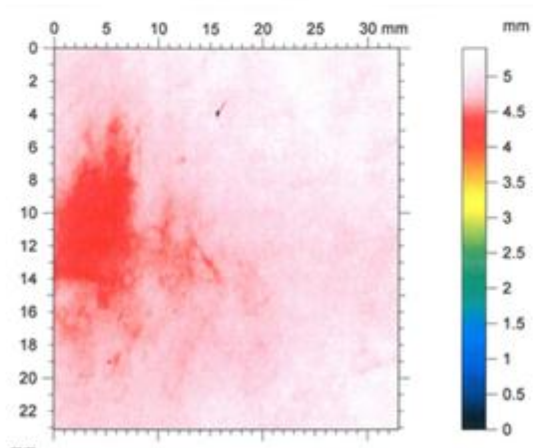
Sa = 0.212 mm
Sp = 0.615 mm
Sv = 4.99 mm
Sku = 10.6
St = 5.6 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0362 mm
Spk = 0.024 mm
Svk = 0.0199 mm
Sr1 = 10.3 %
Sr2 = 84 %

Functional Parameters

Vvv = 0.0303 mm3/mm2



Parameters calculated on the surface RB1 1-10-2011

Amplitude Parameters

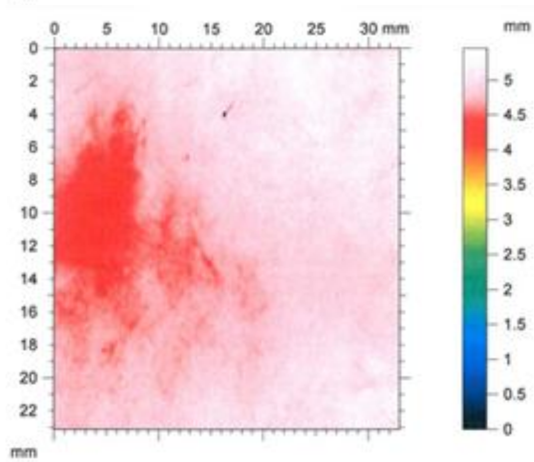
Sa = 0.193 mm
Sp = 0.513 mm
Sv = 4.89 mm
Sku = 14.1
St = 5.4 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0352 mm
Spk = 0.0237 mm
Svk = 0.0208 mm
Sr1 = 12.4 %
Sr2 = 84.2 %

Functional Parameters

Vvv = 0.0336 mm3/mm2



Parameters calculated on the surface RB1 3-11-2011

Amplitude Parameters

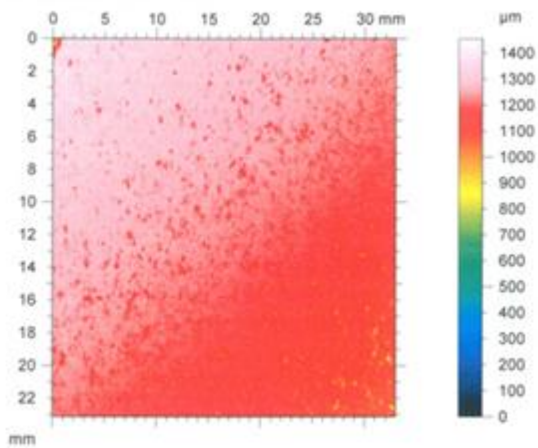
Sa = 0.204 mm
Sp = 0.57 mm
Sv = 4.89 mm
Sku = 11.3
St = 5.46 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0385 mm
Spk = 0.0229 mm
Svk = 0.0206 mm
Sr1 = 10.4 %
Sr2 = 86.1 %

Functional Parameters

Vvv = 0.0343 mm3/mm2



Parameters calculated on the surface RL1 12-9-2010

Amplitude Parameters

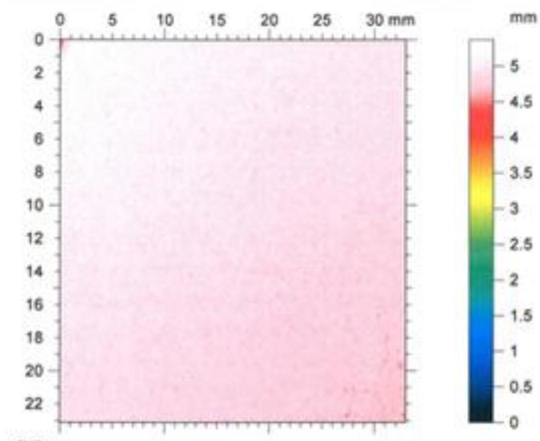
```
Sa   = 61 µm
Sp   = 229 µm
Sv   = 1225 µm
Sku  = 3
St   = 1455 µm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 23.1 µm
Spk  = 13.3 µm
Svk  = 36 µm
Sr1  = 9.63 %
Sr2  = 81.6 %
```

Functional Parameters

```
Vvv  = 0.00848 mm3/mm2
```



Parameters calculated on the surface RL1 1-7-2011

Amplitude Parameters

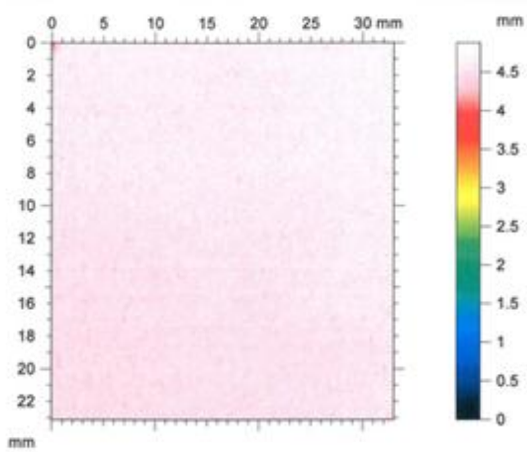
```
Sa   = 0.112 mm
Sp   = 0.367 mm
Sv   = 5 mm
Sku  = 5.54
St   = 5.37 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 0.0228 mm
Spk  = 0.0147 mm
Svk  = 0.0338 mm
Sr1  = 9.07 %
Sr2  = 81.5 %
```

Functional Parameters

```
Vvv  = 0.0153 mm3/mm2
```



Parameters calculated on the surface RL1 3-10-2011

Amplitude Parameters

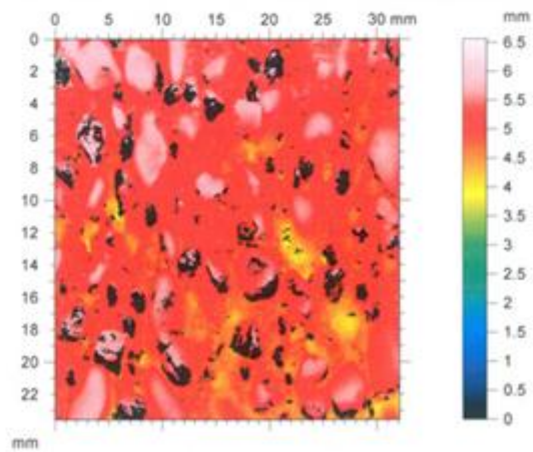
```
Sa   = 0.0547 mm
Sp   = 0.251 mm
Sv   = 4.63 mm
Sku  = 59.9
St   = 4.88 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 0.0223 mm
Spk  = 0.0148 mm
Svk  = 0.0346 mm
Sr1  = 10.4 %
Sr2  = 80.6 %
```

Functional Parameters

```
Vvv  = 0.00753 mm3/mm2
```



Parameters calculated on the surface RC1 12-10-2010

Amplitude Parameters

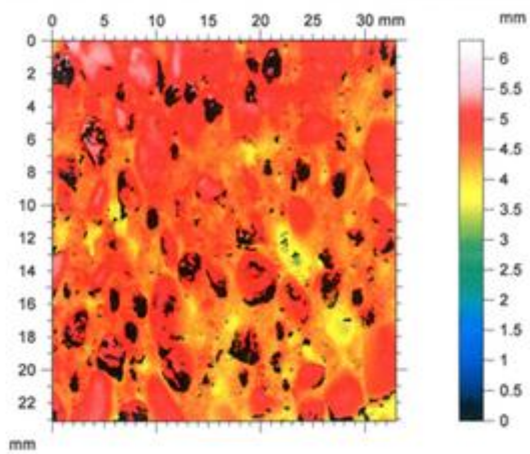
```
Sa   = 0.733 mm
Sp   = 1.81 mm
Sv   = 4.76 mm
Sku  = 10.7
St   = 6.56 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 0.16 mm
Spk  = 1.03 mm
Svk  = 1.8 mm
Sr1  = 22.1 %
Sr2  = 84.9 %
```

Functional Parameters

```
Vvv  = 0.371 mm3/mm2
```



Parameters calculated on the surface RC1 1-10-2011

Amplitude Parameters

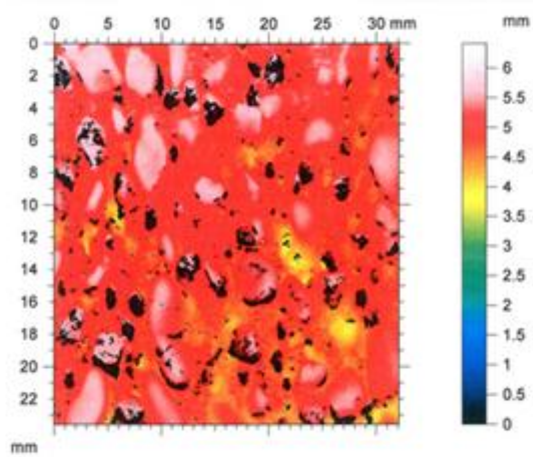
```
Sa   = 0.634 mm
Sp   = 2.02 mm
Sv   = 4.29 mm
Sku  = 11.2
St   = 6.31 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 0.142 mm
Spk  = 0.884 mm
Svk  = 1.54 mm
Sr1  = 22.8 %
Sr2  = 84.5 %
```

Functional Parameters

```
Vvv  = 0.313 mm3/mm2
```



Parameters calculated on the surface RC1 3-16-11

Amplitude Parameters

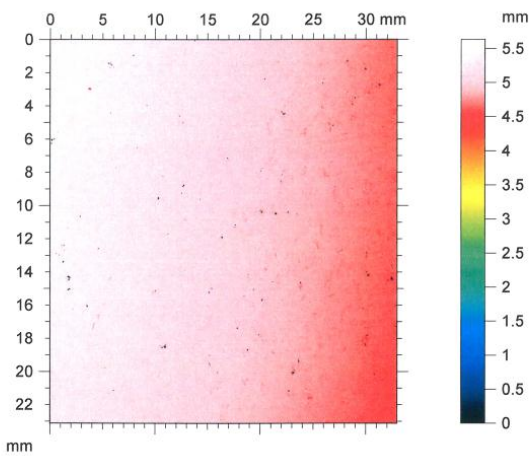
```
Sa   = 0.682 mm
Sp   = 1.73 mm
Sv   = 4.68 mm
Sku  = 11.4
St   = 6.42 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk   = 0.155 mm
Spk  = 0.896 mm
Svk  = 1.88 mm
Sr1  = 24.5 %
Sr2  = 86.2 %
```

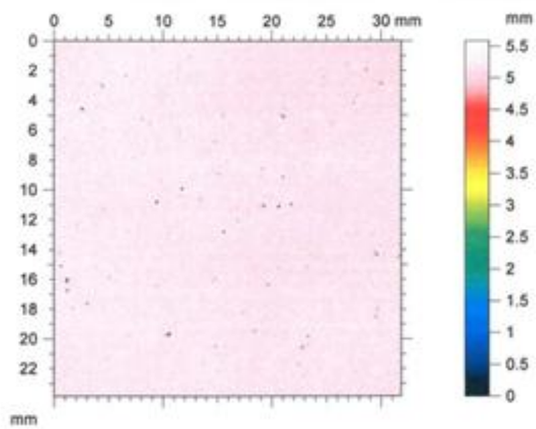
Functional Parameters

```
Vvv  = 0.339 mm3/mm2
```



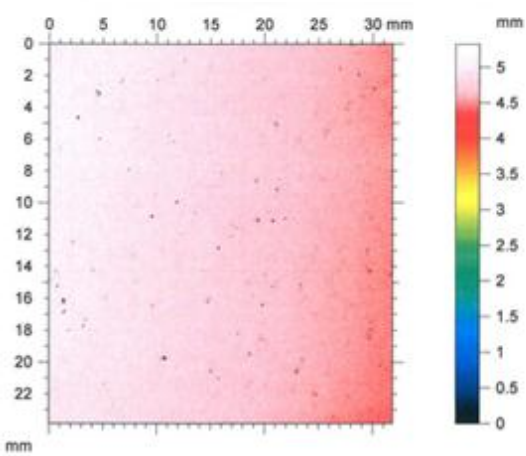
Parameters calculated on the surface RG1 12-7-2010

```
Amplitude Parameters

   Sa     = 0.216 mm
   Sp     = 0.58 mm
   Sv     = 5.05 mm
   Sku    = 62.3
   St     = 5.63 mm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 0.0173 mm
   Spk    = 0.0386 mm
   Svk    = 0.0375 mm
   Sr1    = 12.7 %
   Sr2    = 77.7 %

Functional Parameters

   Vvv    = 0.0224 mm3/mm2
```



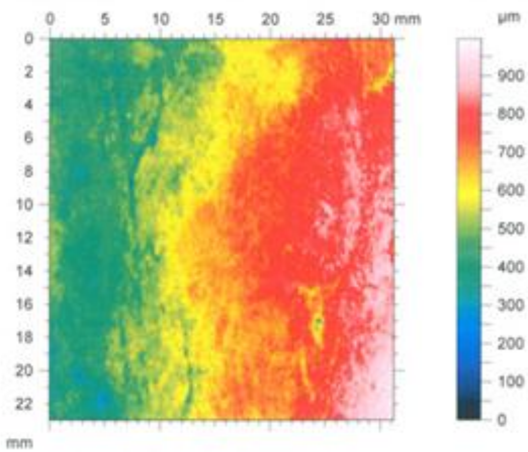
Parameters calculated on the surface rg1 1-7-2011

```
Amplitude Parameters

   Sa     = 0.0378 mm
   Sp     = 0.335 mm
   Sv     = 5.25 mm
   Sku    = 1617
   St     = 5.59 mm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 0.017 mm
   Spk    = 0.0373 mm
   Svk    = 0.034 mm
   Sr1    = 13.3 %
   Sr2    = 79.4 %

Functional Parameters

   Vvv    = 0.00853 mm3/mm2
```



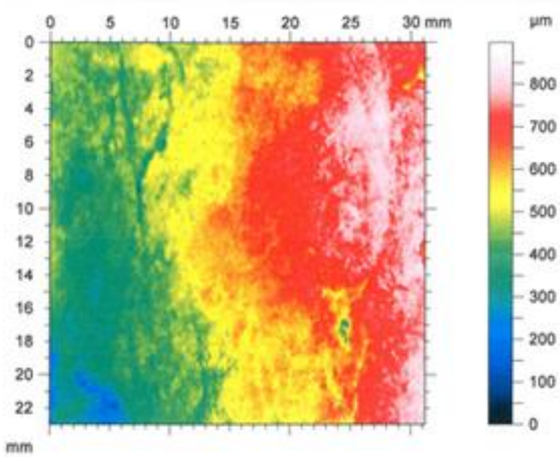
Parameters calculated on the surface RG1 3-11-2011

```
Amplitude Parameters

   Sa     = 0.171 mm
   Sp     = 0.5 mm
   Sv     = 4.83 mm
   Sku    = 128
   St     = 5.33 mm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 0.0107 mm
   Spk    = 0.0336 mm
   Svk    = 0.035 mm
   Sr1    = 23.6 %
   Sr2    = 69.3 %

Functional Parameters

   Vvv    = 0.0182 mm3/mm2
```



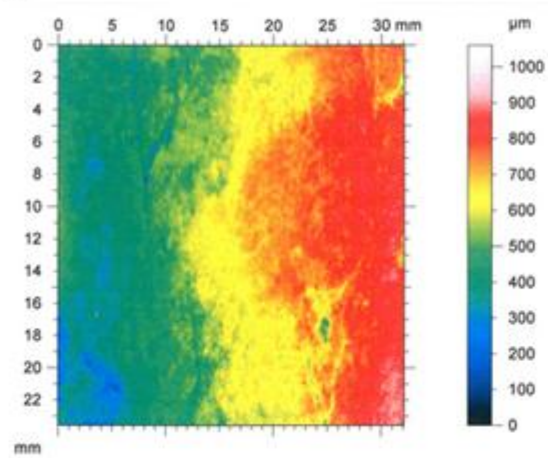
Parameters calculated on the surface GB1 12-7-2010

```
Amplitude Parameters

  Sa     = 134 µm
  Sp     = 377 µm
  Sv     = 620 µm
  Sku    = 1.87
  St     = 997 µm

Functional Parameters, gaussian filter, 0.8 mm

  Sk     = 34.5 µm
  Spk    = 15.1 µm
  Svk    = 18.5 µm
  Sr1    = 10.1 %
  Sr2    = 88.9 %

Functional Parameters

  Vvv    = 0.00915 mm3/mm2
```



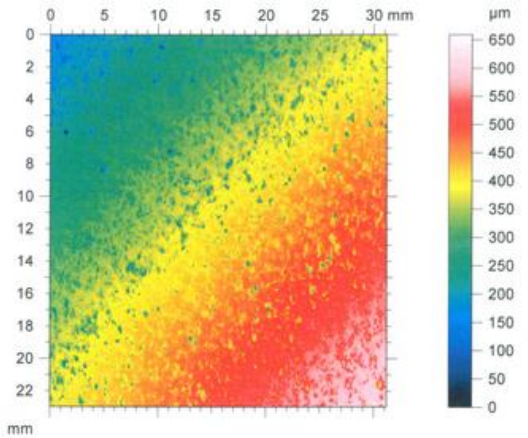
Parameters calculated on the surface GB1 1-10-2011

```
Amplitude Parameters

  Sa     = 129 µm
  Sp     = 333 µm
  Sv     = 565 µm
  Sku    = 2
  St     = 898 µm

Functional Parameters, gaussian filter, 0.8 mm

  Sk     = 33.4 µm
  Spk    = 15.7 µm
  Svk    = 17.6 µm
  Sr1    = 10.7 %
  Sr2    = 89 %

Functional Parameters

  Vvv    = 0.013 mm3/mm2
```



Parameters calculated on the surface GB1 3-11-2011

```
Amplitude Parameters

  Sa     = 140 µm
  Sp     = 467 µm
  Sv     = 593 µm
  Sku    = 1.9
  St     = 1060 µm

Functional Parameters, gaussian filter, 0.8 mm

  Sk     = 33.8 µm
  Spk    = 16 µm
  Svk    = 17.3 µm
  Sr1    = 10.5 %
  Sr2    = 89.1 %

Functional Parameters

  Vvv    = 0.0121 mm3/mm2
```



Parameters calculated on the surface GL1 12-9-2010

Amplitude Parameters

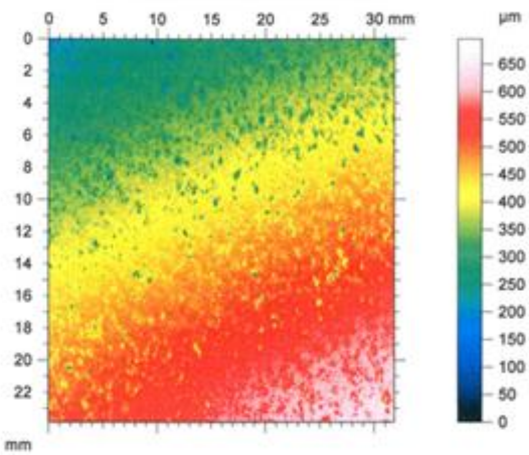
Sa	= 79.5 µm
Sp	= 273 µm
Sv	= 386 µm
Sku	= 2.5
St	= 659 µm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 27 µm
Spk	= 11.6 µm
Svk	= 34.9 µm
Sr1	= 7.69 %
Sr2	= 81.2 %

Functional Parameters

Vvv = 0.0106 mm3/mm2



Parameters calculated on the surface GL1 1-7-2011

Amplitude Parameters

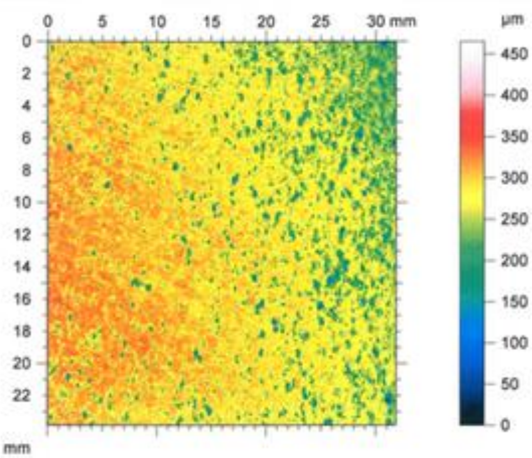
Sa	= 82.7 µm
Sp	= 263 µm
Sv	= 434 µm
Sku	= 2.3
St	= 696 µm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 30.7 µm
Spk	= 10.7 µm
Svk	= 28.2 µm
Sr1	= 7.63 %
Sr2	= 83.4 %

Functional Parameters

Vvv = 0.00972 mm3/mm2



Parameters calculated on the surface GL1 3-10-2011

Amplitude Parameters

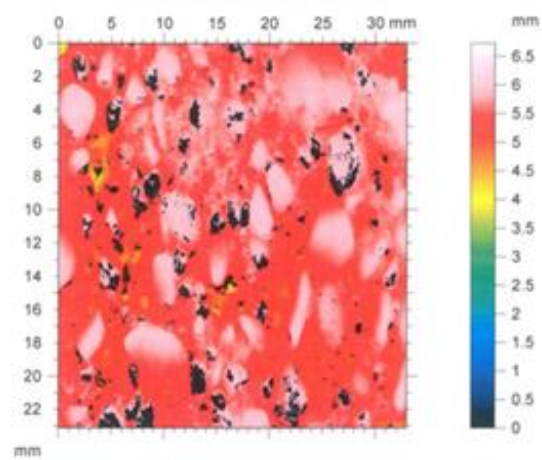
Sa	= 25.1 µm
Sp	= 187 µm
Sv	= 278 µm
Sku	= 5.75
St	= 465 µm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 33.1 µm
Spk	= 11.1 µm
Svk	= 28.4 µm
Sr1	= 7.32 %
Sr2	= 83.5 %

Functional Parameters

Vvv = 0.00523 mm3/mm2



Parameters calculated on the surface GC1 12-10-2010

Amplitude Parameters

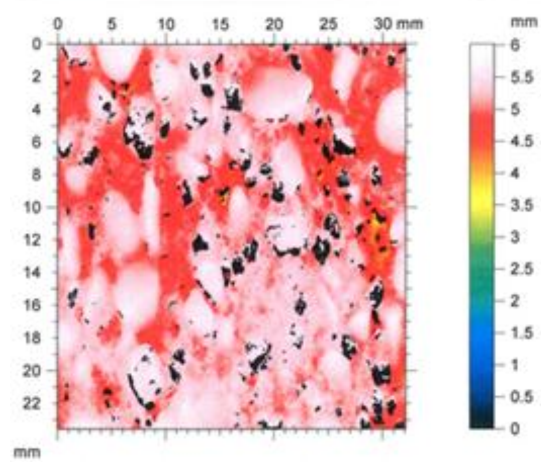
Sa	= 0.621 mm
Sp	= 1.49 mm
Sv	= 5.23 mm
Sku	= 14.7
St	= 6.73 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 0.11 mm
Spk	= 0.882 mm
Svk	= 1.49 mm
Sr1	= 24.6 %
Sr2	= 83.4 %

Functional Parameters

Vvv = 0.31 mm3/mm2



Parameters calculated on the surface GC1 1-10-2011

Amplitude Parameters

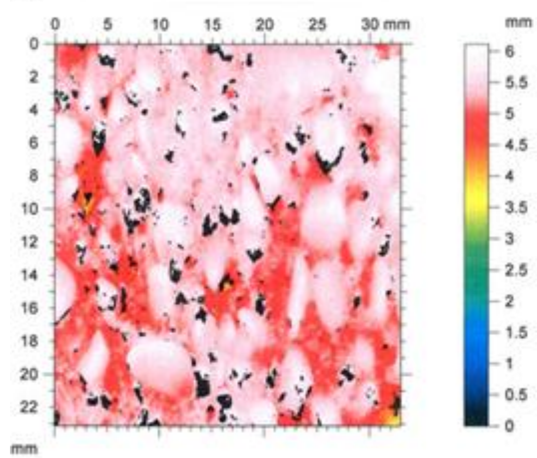
Sa	= 0.58 mm
Sp	= 1.07 mm
Sv	= 4.96 mm
Sku	= 15.1
St	= 6.02 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 0.126 mm
Spk	= 0.864 mm
Svk	= 1.53 mm
Sr1	= 22.7 %
Sr2	= 84.9 %

Functional Parameters

Vvv = 0.284 mm3/mm2



Parameters calculated on the surface GC1 3-16-11

Amplitude Parameters

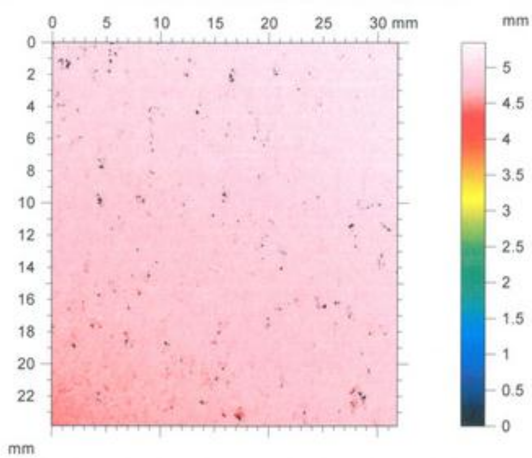
Sa	= 0.522 mm
Sp	= 0.916 mm
Sv	= 5.2 mm
Sku	= 18.8
St	= 6.12 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk	= 0.111 mm
Spk	= 0.784 mm
Svk	= 1.26 mm
Sr1	= 22.1 %
Sr2	= 83.6 %

Functional Parameters

Vvv = 0.241 mm3/mm2



Parameters calculated on the surface GG1 12-7-2010

Amplitude Parameters

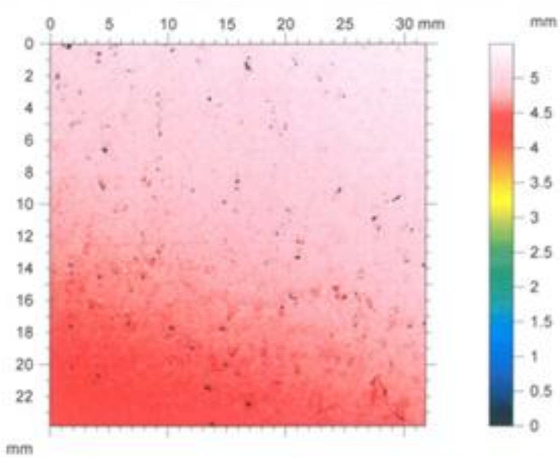
```
Sa     = 0.133 mm
Sp     = 0.494 mm
Sv     = 4.85 mm
Sku    = 206
St     = 5.35 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk     = 0.0434 mm
Spk    = 0.112 mm
Svk    = 0.176 mm
Sr1    = 16.2 %
Sr2    = 84.6 %
```

Functional Parameters

```
Vvv    = 0.0283 mm3/mm2
```



Parameters calculated on the surface GG1 1-10-2011

Amplitude Parameters

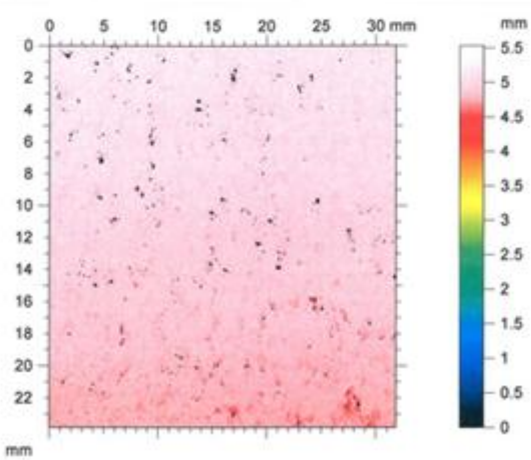
```
Sa     = 0.219 mm
Sp     = 0.695 mm
Sv     = 4.8 mm
Sku    = 98
St     = 5.49 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk     = 0.0456 mm
Spk    = 0.116 mm
Svk    = 0.179 mm
Sr1    = 15.2 %
Sr2    = 84 %
```

Functional Parameters

```
Vvv    = 0.0352 mm3/mm2
```



Parameters calculated on the surface GG1 3-16-2011

Amplitude Parameters

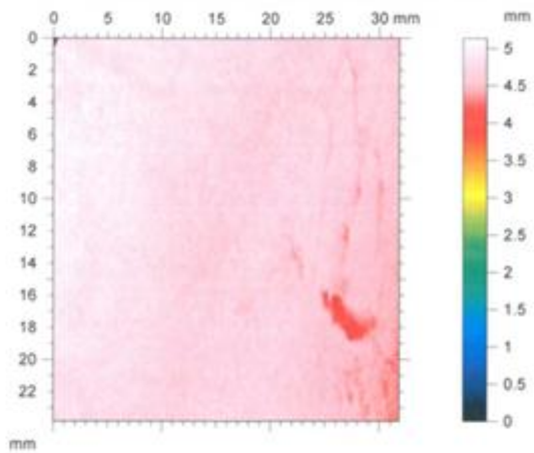
```
Sa     = 0.149 mm
Sp     = 0.56 mm
Sv     = 4.97 mm
Sku    = 167
St     = 5.53 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk     = 0.0525 mm
Spk    = 0.144 mm
Svk    = 0.272 mm
Sr1    = 15.5 %
Sr2    = 87 %
```

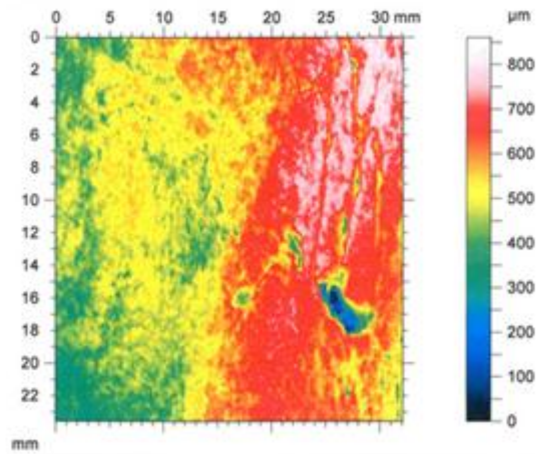
Functional Parameters

```
Vvv    = 0.0311 mm3/mm2
```



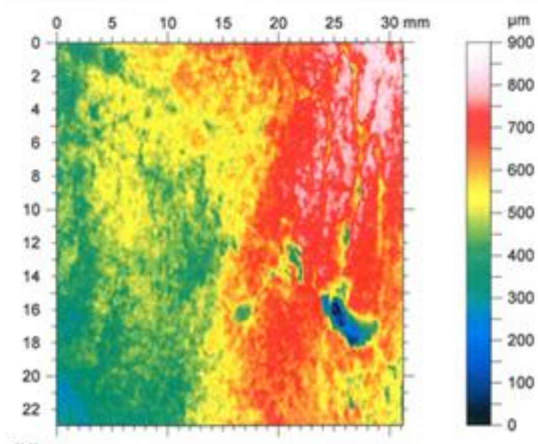
Parameters calculated on the surface WB1 12-10-2010

```
Amplitude Parameters

   Sa     = 0.123 mm
   Sp     = 0.348 mm
   Sv     = 4.79 mm
   Sku    = 47.8
   St     = 5.14 mm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 0.0413 mm
   Spk    = 0.0181 mm
   Svk    = 0.0231 mm
   Sr1    = 9.68 %
   Sr2    = 89 %

Functional Parameters

   Vvv    = 0.0296 mm3/mm2
```



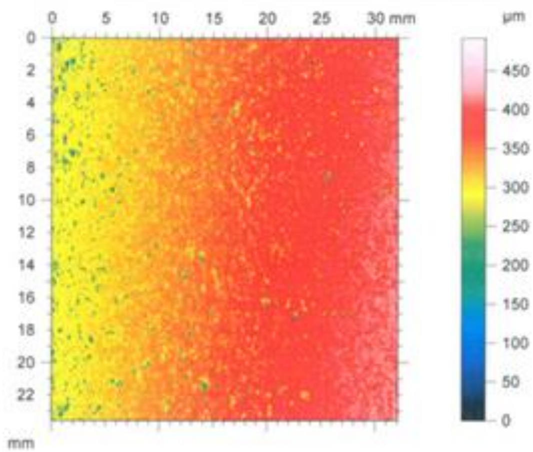
Parameters calculated on the surface WB1 1-10-2011

```
Amplitude Parameters

   Sa     = 95.1 µm
   Sp     = 304 µm
   Sv     = 557 µm
   Sku    = 2.79
   St     = 861 µm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 42.4 µm
   Spk    = 18.4 µm
   Svk    = 23.6 µm
   Sr1    = 9.61 %
   Sr2    = 88.8 %

Functional Parameters

   Vvv    = 0.0107 mm3/mm2
```



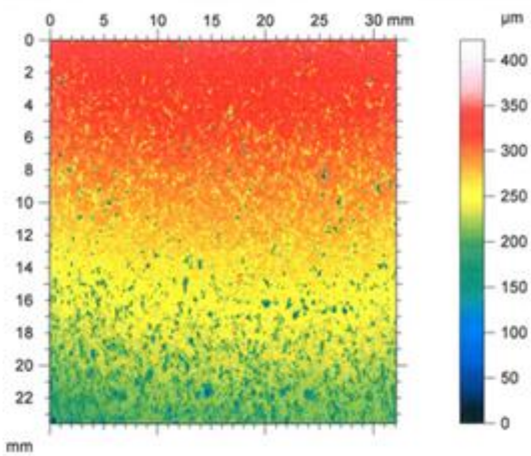
Parameters calculated on the surface WB1 3-11-2011

```
Amplitude Parameters

   Sa     = 103 µm
   Sp     = 347 µm
   Sv     = 554 µm
   Sku    = 2.73
   St     = 901 µm

Functional Parameters, gaussian filter, 0.8 mm

   Sk     = 43.1 µm
   Spk    = 18.6 µm
   Svk    = 23.8 µm
   Sr1    = 9.76 %
   Sr2    = 88.8 %

Functional Parameters

   Vvv    = 0.0112 mm3/mm2
```



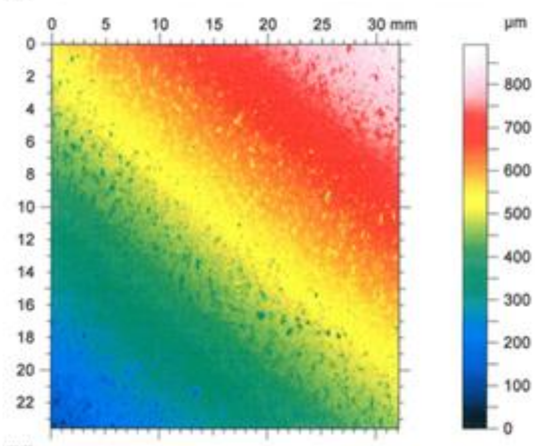
Parameters calculated on the surface WL1 12-9-2010

```
Amplitude Parameters

   Sa      = 35 µm
   Sp      = 145 µm
   Sv      = 346 µm
   Sku     = 2.91
   St      = 492 µm

Functional Parameters, gaussian filter, 0.8 mm

   Sk      = 20.4 µm
   Spk     = 9.92 µm
   Svk     = 29.3 µm
   Sr1     = 8.42 %
   Sr2     = 80.9 %

Functional Parameters

   Vvv     = 0.00447 mm3/mm2
```



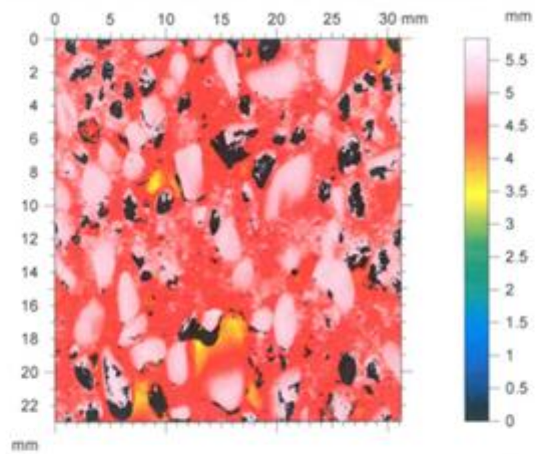
Parameters calculated on the surface WL1 1-7-2011

```
Amplitude Parameters

   Sa      = 37.5 µm
   Sp      = 155 µm
   Sv      = 267 µm
   Sku     = 2.85
   St      = 423 µm

Functional Parameters, gaussian filter, 0.8 mm

   Sk      = 19.3 µm
   Spk     = 9.35 µm
   Svk     = 28.3 µm
   Sr1     = 8.59 %
   Sr2     = 80.9 %

Functional Parameters

   Vvv     = 0.00444 mm3/mm2
```



Parameters calculated on the surface WL1 3-10-2011

```
Amplitude Parameters

   Sa      = 124 µm
   Sp      = 386 µm
   Sv      = 507 µm
   Sku     = 2.43
   St      = 893 µm

Functional Parameters, gaussian filter, 0.8 mm

   Sk      = 19.8 µm
   Spk     = 9.72 µm
   Svk     = 28.8 µm
   Sr1     = 8.25 %
   Sr2     = 81.1 %

Functional Parameters

   Vvv     = 0.0162 mm3/mm2
```



Parameters calculated on the surface WC1 12-10-10

Amplitude Parameters

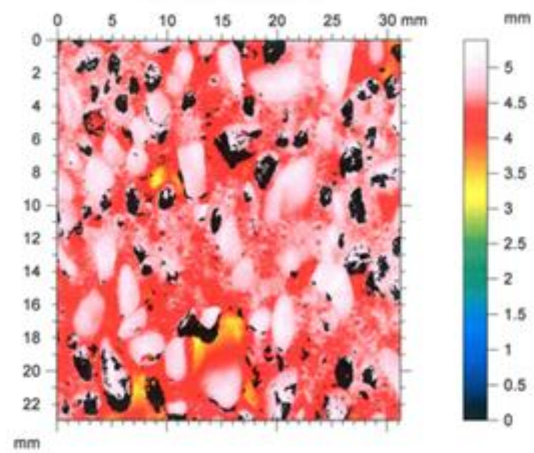
```
Sa    = 0.782 mm
Sp    = 1.36 mm
Sv    = 4.47 mm
Sku   = 9.16
St    = 5.83 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk    = 0.182 mm
Spk   = 0.984 mm
Svk   = 1.7 mm
Sr1   = 21.1 %
Sr2   = 85.2 %
```

Functional Parameters

```
Vvv   = 0.406 mm3/mm2
```



Parameters calculated on the surface WC1 1-10-2011

Amplitude Parameters

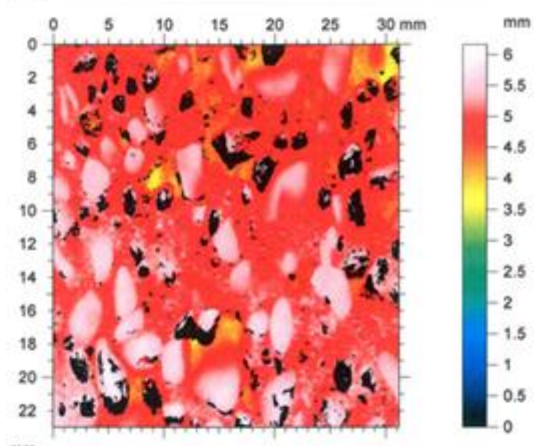
```
Sa    = 0.761 mm
Sp    = 1.18 mm
Sv    = 4.21 mm
Sku   = 8.79
St    = 5.39 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk    = 0.158 mm
Spk   = 0.89 mm
Svk   = 1.56 mm
Sr1   = 23.6 %
Sr2   = 84 %
```

Functional Parameters

```
Vvv   = 0.39 mm3/mm2
```



Parameters calculated on the surface WC1 3-16-11

Amplitude Parameters

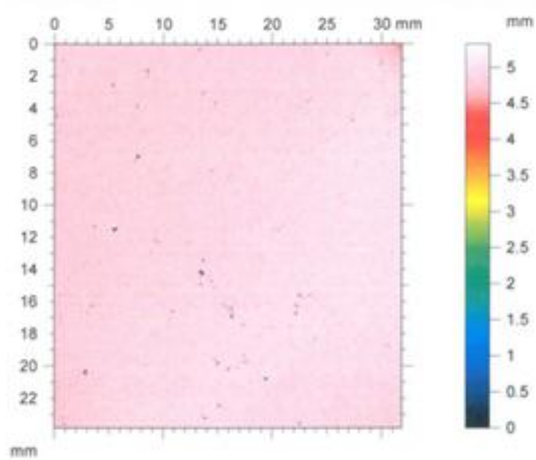
```
Sa    = 0.839 mm
Sp    = 1.57 mm
Sv    = 4.59 mm
Sku   = 8.58
St    = 6.17 mm
```

Functional Parameters, gaussian filter, 0.8 mm

```
Sk    = 0.188 mm
Spk   = 1.01 mm
Svk   = 1.89 mm
Sr1   = 22.7 %
Sr2   = 85.2 %
```

Functional Parameters

```
Vvv   = 0.429 mm3/mm2
```



Parameters calculated on the surface WG1 12-9-2010

Amplitude Parameters

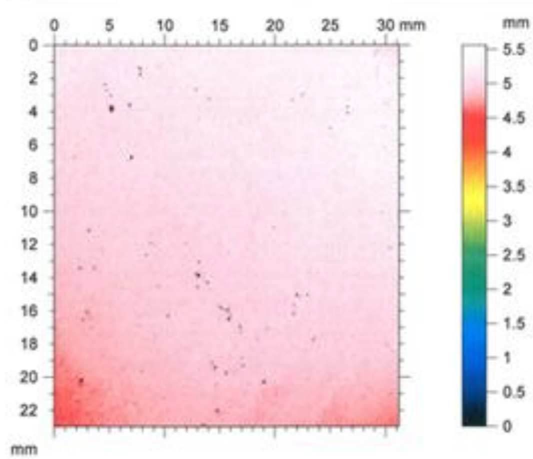
Sa = 0.0619 mm
Sp = 0.337 mm
Sv = 4.99 mm
Sku = 887
St = 5.33 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0117 mm
Spk = 0.0365 mm
Svk = 0.0368 mm
Sr1 = 22.2 %
Sr2 = 71 %

Functional Parameters

Vvv = 0.0121 mm3/mm2



Parameters calculated on the surface WG1 1-7-2011

Amplitude Parameters

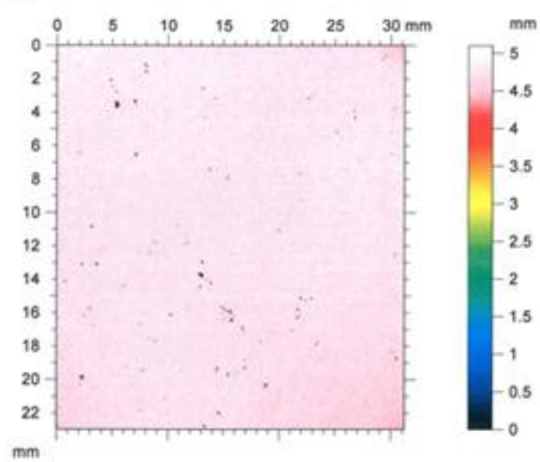
Sa = 0.155 mm
Sp = 0.498 mm
Sv = 5.07 mm
Sku = 185
St = 5.56 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0188 mm
Spk = 0.0454 mm
Svk = 0.0534 mm
Sr1 = 15.1 %
Sr2 = 82.1 %

Functional Parameters

Vvv = 0.0216 mm3/mm2



Parameters calculated on the surface WG1 3-10-2011

Amplitude Parameters

Sa = 0.0513 mm
Sp = 0.32 mm
Sv = 4.78 mm
Sku = 785
St = 5.1 mm

Functional Parameters, gaussian filter, 0.8 mm

Sk = 0.0176 mm
Spk = 0.0484 mm
Svk = 0.0505 mm
Sr1 = 15.7 %
Sr2 = 76.9 %

Functional Parameters

Vvv = 0.0187 mm3/mm2

Sample ID	Core Roughness Depth (Sk) in mm			
	Before Treatment; 0 Hours)	Midpoint (337 Hours)	After Treatment (800 Hours)	Overall Change in Sk
RB1	0.0362	0.0352	0.0385	0.0023
RL1	0.0231	0.0228	0.0223	-0.0008
RC1	0.16	0.142	0.155	-0.005
RG1	0.0173	0.017	0.0107	-0.0066
GB1	0.0345	0.0334	0.0338	-0.0007
GL1	0.027	0.0307	0.0331	0.0061
GC1	0.11	0.126	0.111	0.001
GG1	0.0434	0.0456	0.0525	0.0091
WB1	0.0413	0.0424	0.0431	0.0018
WL1	0.0204	0.0193	0.0198	-0.0006
WC1	0.182	0.158	0.188	0.006
WG1	0.0117	0.0188	0.0176	0.0059

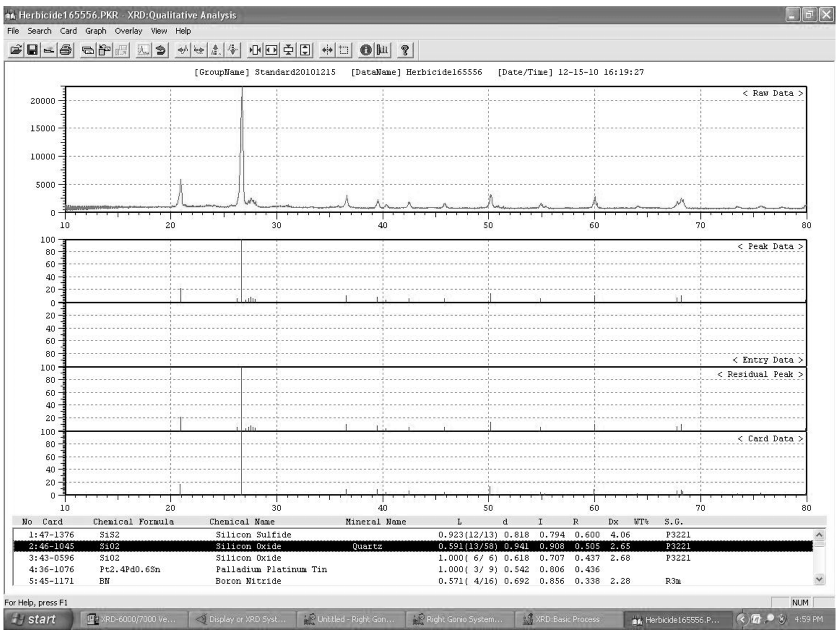
Sample ID	Reduced Peaks Height (Spk) in mm			
	Before Treatment; 0 Hours)	Midpoint (337 Hours)	After Treatment (800 Hours)	Overall Change in Spk
RB1	0.024	0.0237	0.0229	-0.0011
RL1	0.0133	0.0147	0.0148	0.0015
RC1	1.03	0.884	0.896	-0.134
RG1	0.0386	0.0373	0.0336	-0.005
GB1	0.0151	0.0157	0.016	0.0009
GL1	0.0116	0.0107	0.0111	-0.0005
GC1	0.882	0.864	0.784	-0.098
GG1	0.112	0.116	0.144	0.032
WB1	0.0181	0.0184	0.0186	0.0005
WL1	0.00992	0.00935	0.00972	-0.0002
WC1	0.984	0.89	1.01	0.026
WG1	0.0365	0.0454	0.0484	0.0119

Sample ID	Reduced Valley Depth (Svk) in mm			
	Before Treatment; 0 Hours)	Midpoint (337 Hours)	After Treatment (800 Hours)	Overall Change in Svk
RB1	0.0199	0.0208	0.0206	0.0007
RL1	0.036	0.0338	0.0346	-0.0014
RC1	1.8	1.54	1.88	0.08
RG1	0.0375	0.034	0.035	-0.0025
GB1	0.0185	0.0176	0.0173	-0.0012
GL1	0.0349	0.0282	0.0284	-0.0065
GC1	1.49	1.53	1.26	-0.23
GG1	0.176	0.179	0.272	0.096
WB1	0.0231	0.0236	0.0238	0.0007
WL1	0.0293	0.0283	0.0288	-0.0005
WC1	1.7	1.56	1.89	0.19
WG1	0.0368	0.0534	0.0505	0.0137

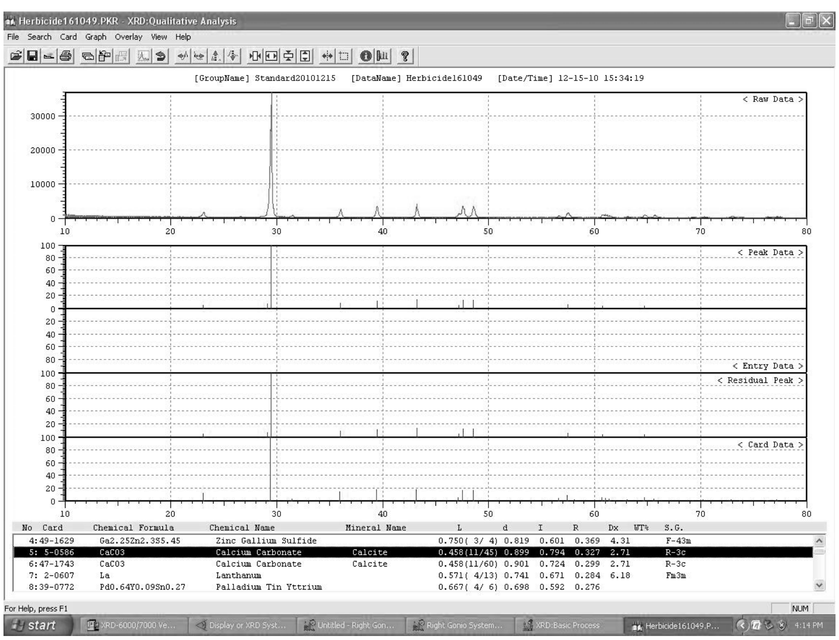
APPENDIX F

X-RAY DEFRACTION DATA

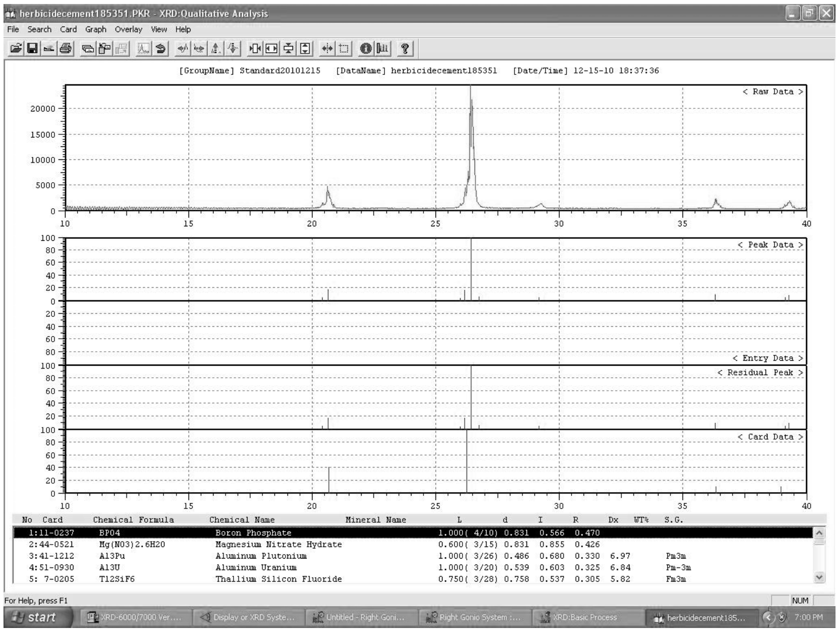
Brick before treatment:



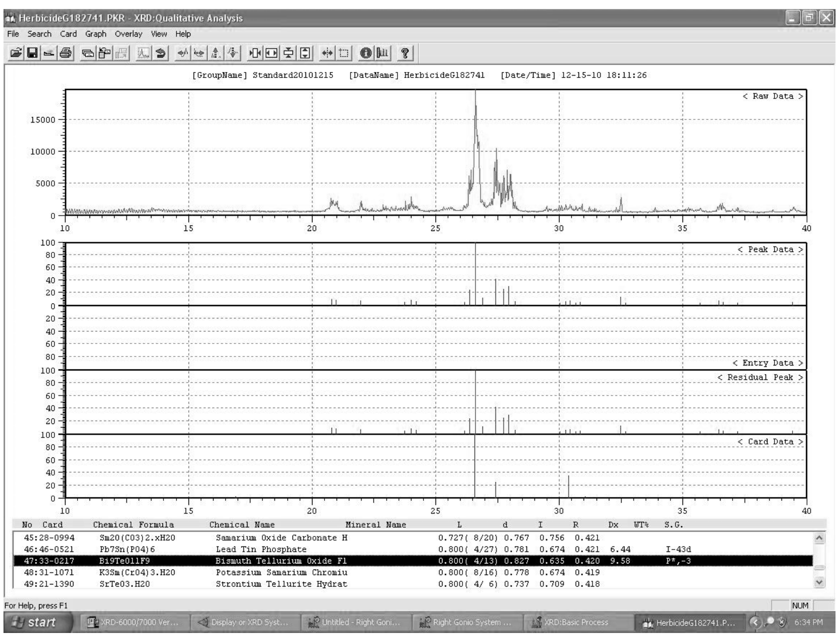
Limestone before treatment:



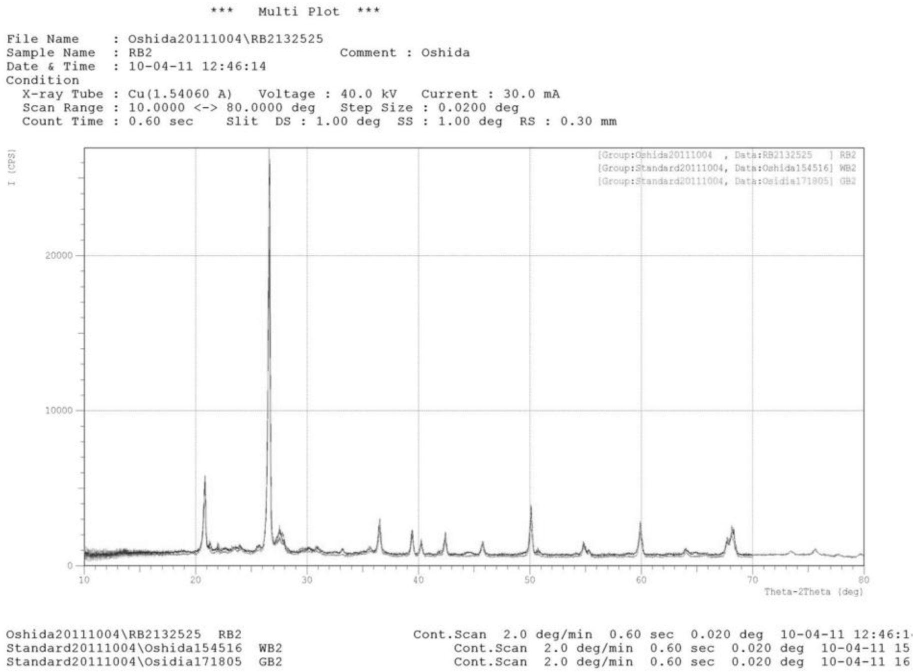
Concrete before treatment:



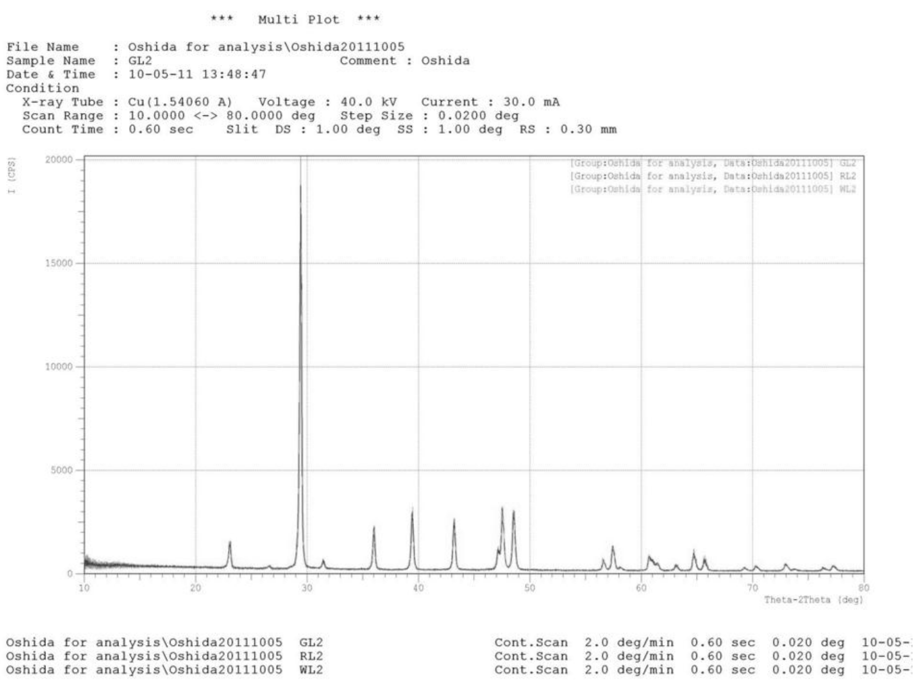
Granite before treatment:



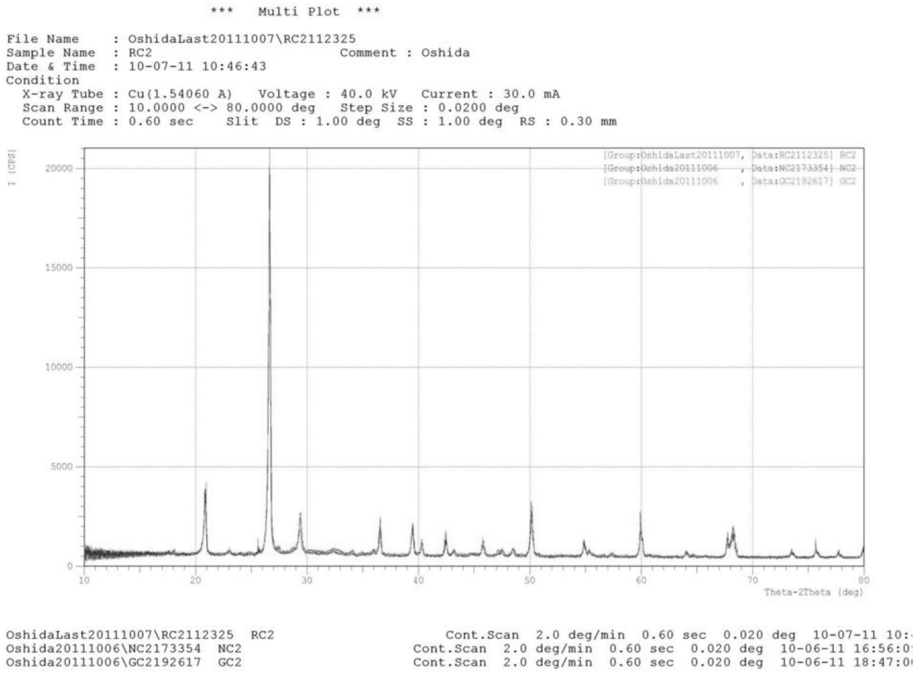
The overlaid XRD spectrum of samples RB2, GB2, and WB2:



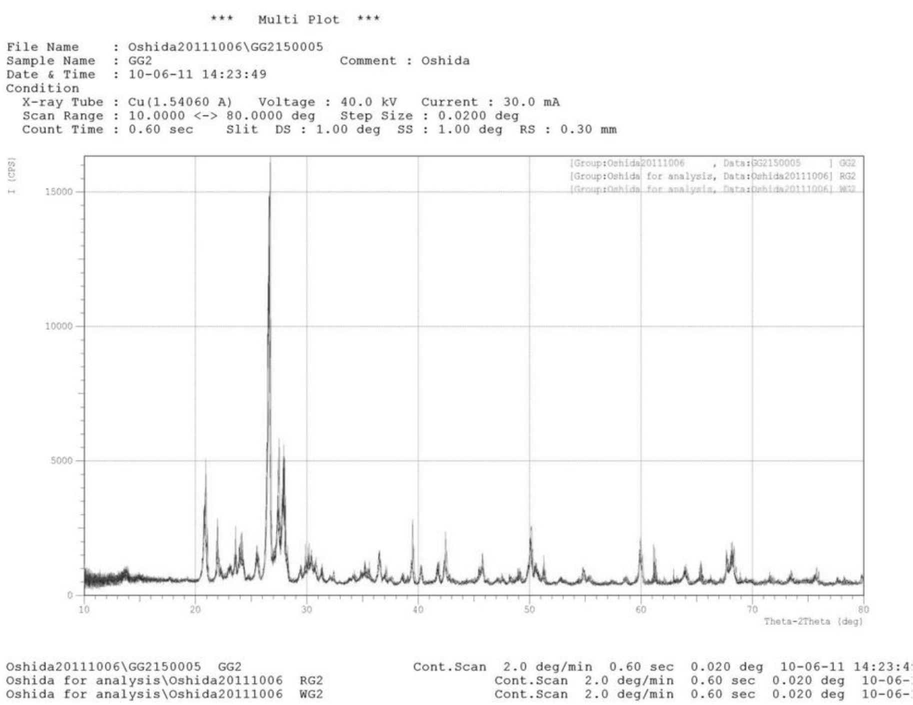
The overlaid XRD spectrum of samples RL2, GL2, and WL2:



The overlaid XRD spectrum of samples RC2, GC2, and WC2:



The overlaid XRD spectrum of samples RG2, GG2, and WG2:

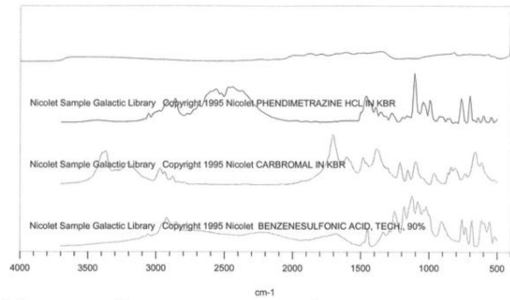


APPENDIX G

FT-IR DATA

No treatment, brick sample

HERB_NB1.SPC Sample 001 By Administrator Date Thursday, March 17 2011



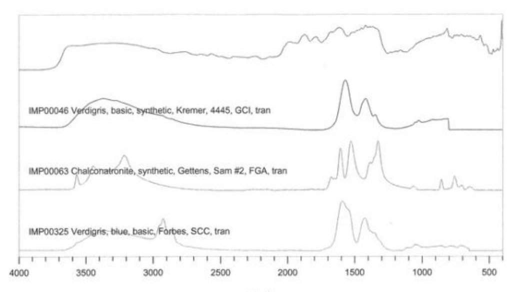
Hit #	Quality	Library	Memo
1	**0.662982**	**nicsam.lib**	**PHENDIMETRAZINE HCL IN KBR**
2	**0.678251**	**nicsam.lib**	**CARBROMAL IN KBR**
3	**0.686854**	**nicsam.lib**	**BENZENESULFONIC ACID, TECH., 90%**
4	0.715445	nicsam.lib	PHENYLPHOSPHONIC ACID, 98%
5	0.720945	nicsam.lib	1-(1-(2-THIENYL)CYCLOHEXYL)MORPHO
6	0.73207	nicsam.lib	5-AMINO-2-METHYLBENZOTHIAZOLE DIHYDROCHLORIDE
7	0.739832	nicsam.lib	NICOTINE HCL IN KBR
8	0.740541	nicsam.lib	HOMATROPINE HYDROBROMIDE IN KBR
9	0.747841	epavpst.lib	WATER DISTILLED
10	0.753236	nicsam.lib	ETHANESULFONIC ACID
11	0.753582	nicsam.lib	PETHIDINE HCL IN KBR
12	0.761968	nicsam.lib	BENZPHETAMINE HCL IN KBR
13	0.764146	nicsam.lib	1(1,2-THIENYL)CYCLOHEXYLPIPERIDIN
14	0.787232	nicsam.lib	ERYTHROMYCIN STEARATE IN KBR
15	0.789243	nicsam.lib	AMITRIPTYLINE HCL IN KBR
16	0.790052	nicsam.lib	PENICILLIN G POTASSIUM IN KBR
17	0.790599	nicsam.lib	TCP PYRROLIDINE ANALOG HCL IN KBR
18	0.790791	nicsam.lib	LSD TARTRATE IN KBR
19	0.793966	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
20	0.797579	nicsam.lib	DIPHENHYDRAMINE HCL IN KBR

Search Options Used

Search Type:	Spectrum
[Spectrum Search]	
Algorithm:	Correlation
[Preprocessing]	
Baselining:	On
Region Masking:	Off
[Libraries]	
Libraries:	Local
[Spectrum]	
Spectrum:	C:\pel_data\Caitelin\HERB_NB1.SPC (1 of 1)

Roundup®, brick sample

HERB-RB2.SPC Sample 013 By Administrator Date Tuesday, March 15 2011



Hit #	Quality	Library	Memo
1	**0.578415**	**Ncptt.lib**	**IMP00046 Verdigris, basic, synthetic, Kremer, 4445, GCI, tra**
2	**0.602437**	**Ncptt.lib**	**IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA**
3	**0.609777**	**Ncptt.lib**	**IMP00325 Verdigris, blue, basic, Forbes, SCC, tran**
4	0.667248	nicsam.lib	ERYTHROMYCIN STEARATE IN KBR
5	0.671403	nicsam.lib	CARBROMAL IN KBR
6	0.675736	Ncptt.lib	IMP00264 Artinite, UD, #D-1358, PMA, tran
7	0.678364	Ncptt.lib	IMP00289 Aurichalcite, Mapimi, Durango, MX, SI-NMNH, #1246
8	0.679901	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
9	0.684219	Ncptt.lib	IMP00152 Verdigris, neutral, Forbes: Buckner, PMA# K-52, PM/
10	0.686539	nicsam.lib	BENZENESULFONIC ACID, TECH., 90%
11	0.688182	Ncptt.lib	IMP00098 Copper acetate, Cargille, #11-B, PMA, tran
12	0.691198	nicsam.lib	FOLIC ACID IN KBR
13	0.695543	Ncptt.lib	IMP00406 Verdigris, synthetic, UCLA Cons Lab, experiment 6-2
14	0.695578	Ncptt.lib	IMP00274 Malachite, Kremer, PMA, tran
15	0.698749	Ncptt.lib	IMP00465 Artinite, Union Carbide Mine, Calif, US, BM1973503,
16	0.701793	nicsam.lib	LSD TARTRATE IN KBR
17	0.702404	Ncptt.lib	IMP00326 Verdigris, neutral, Forbes: Roberson, 72, SCC, tran
18	0.703083	Ncptt.lib	IMP00317 Malachite, Forbes, SCC, tran
19	0.704973	Ncptt.lib	IMP00147 Verdigris, neutral, Forbes: Hull, PMA# K-51, PMA, tra
20	0.706348	Ncptt.lib	IMP00088 Malachite, Forbes: Hamilton, PMA, tran

Search Options Used

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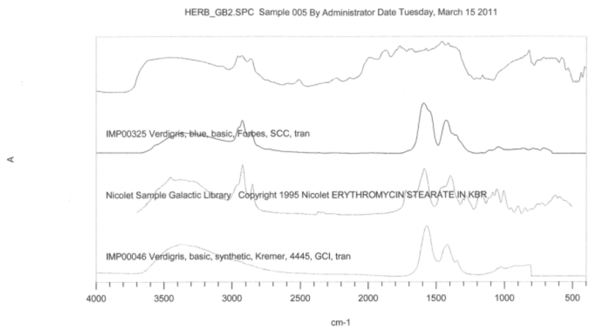
Garlon®4, brick sample



Hit #	Quality	Library	Memo
1	**0.625374**	**Ncptt.lib**	**IMP00325 Verdigris, blue, basic, Forbes, SCC, tran**
2	**0.652622**	**nicsam.lib**	**ERYTHROMYCIN STEARATE IN KBR**
3	**0.655029**	**Ncptt.lib**	**IMP00046 Verdigris, basic, synthetic, Kremer, 4445, GCI, tra**
4	0.669975	nicsam.lib	CARBROMAL IN KBR
5	0.69483	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
6	0.71477	Ncptt.lib	IMP00264 Artinite, UD, #D-1358, PMA, tran
7	0.72306	nicsam.lib	PHENDIMETRAZINE HCL IN KBR
8	0.727591	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
9	0.735772	nicsam.lib	BENZENESULFONIC ACID, TECH., 90%
10	0.738226	nicsam.lib	ETHINAMATE IN KBR
11	0.745791	Ncptt.lib	IMP00289 Aurichalcite, Mapimi, Durango, MX, SI-NMNH, #1246
12	0.747046	Ncptt.lib	IMP00152 Verdigris, neutral, Forbes: Buckner, PMA# K-52, PM
13	0.750766	Ncptt.lib	IMP00274 Malachite, Kremer, PMA, tran
14	0.751688	Ncptt.lib	IMP00098 Copper acetate, Cargille, #11-B, PMA, tran
15	0.753447	nicsam.lib	FOLIC ACID IN KBR
16	0.753919	Ncptt.lib	IMP00317 Malachite, Forbes, SCC, tran
17	0.754362	nicsam.lib	AMPICILLIN TRIHYDRATE IN KBR
18	0.760608	nicsam.lib	BUTYRAMIDE
19	0.761154	cc_samp.lib	FE(II)-DODECANOATE; DODECANOIC ACID, FE(II) SALT
20	0.763614	Ncptt.lib	IMP00147 Verdigris, neutral, Forbes: Hull, PMA# K-51, PMA, tra

Search Options Used

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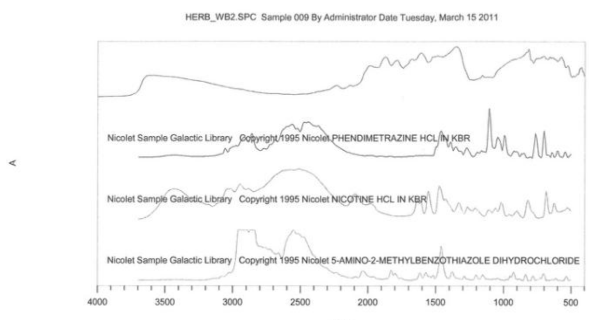
DI water, brick sample



Hit #	Quality	Library	Memo
1	**0.652607**	**nicsam.lib**	**PHENDIMETRAZINE HCL IN KBR**
2	**0.690308**	**nicsam.lib**	**NICOTINE HCL IN KBR**
3	**0.693789**	**nicsam.lib**	**5-AMINO-2-METHYLBENZOTHIAZOLE DIHYDROCHLORIDE**
4	0.700632	nicsam.lib	CARBROMAL IN KBR
5	0.70715	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
6	0.70735	nicsam.lib	PHENYLPHOSPHONIC ACID, 98%
7	0.707877	nicsam.lib	HOMATROPINE HYDROBROMIDE IN KBR
8	0.711409	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
9	0.717691	nicsam.lib	BENZENESULFONIC ACID, TECH., 90%
10	0.720539	nicsam.lib	1-(1-(2-THIENYL)CYCLOHEXYL)MORPHO
11	0.740996	nicsam.lib	1(1,2-THIENYL)CYCLOHEXYLPIPERIDIN
12	0.745534	nicsam.lib	BENZPHETAMINE HCL IN KBR
13	0.747364	nicsam.lib	PETHIDINE HCL IN KBR
14	0.748472	nicsam.lib	ETHANESULFONIC ACID
15	0.749282	Ncptt.lib	IMP00274 Malachite, Kremer, PMA, tran
16	0.750571	Ncptt.lib	IMP00211 Malachite, Mexico, SI-NMNH, #124651, PMA, tran
17	0.755339	Ncptt.lib	IMP00152 Verdigris, neutral, Forbes: Buckner, PMA# K-52, PM
18	0.75702	Ncptt.lib	IMP00098 Copper acetate, Cargille, #11-B, PMA, tran
19	0.764592	epavpst.lib	WATER DISTILLED
20	0.76696	nicsam.lib	TCP PYRROLIDINE ANALOG HCL IN KBR

Search Options Used

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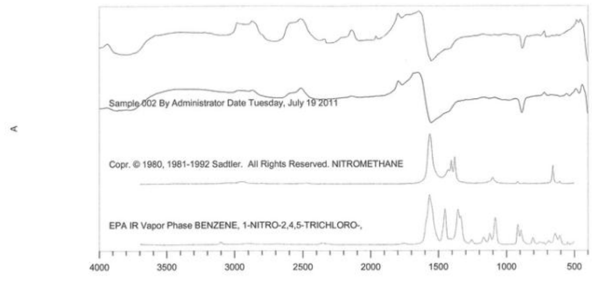
No treatment, limestone sample

HERB_NL1.SPC Sample 002 By Administrator Date Thursday, March 17 2011



Hit #	Quality	Library	Memo
1	**0.161005**	**Ncptt.lib**	**Sample 002 By Administrator Date Tuesday, July 19 2011**
2	**0.782142**	**srldemo.lib**	**NITROMETHANE**
3	**0.784017**	**epavpst.lib**	**BENZENE, 1-NITRO-2,4,5-TRICHLORO-,**
4	0.797185	epavpst.lib	DECANEDIOIC ACID
5	0.79773	srldemo.lib	FORMIC ACID
6	0.799771	epavpst.lib	O-CRESOL, 3-NITRO-,
7	0.800769	epavpst.lib	BENZENE, 1,4-DICHLORO-2-NITRO-,
8	0.80284	epa_gcir.lib	ACETONITRILE
9	0.804231	Ncptt.lib	IMP00025 Gray chalk, calcite with gypsum, Kremer, 5819, GCI,
10	0.805889	epavpst.lib	ADIPIC ACID, MONOMETHYL ESTER
11	0.806655	epavpst.lib	QUINALDINE, 8-NITRO-,
12	0.8086	epavpst.lib	BIPHENYL, 2,2PR-DINITRO-,
13	0.809242	epavpst.lib	OXADIAZOLE, 1,3,4-, 2-(M-FLUORO- PHENYL)-,
14	0.810617	epavpst.lib	P-CRESOL, 3-NITRO-,
15	0.811816	GRAMSSuite.lib	ACETONITRILE
16	0.811871	epavpst.lib	ANILINE, 4-CHLORO-N-METHYL-3- NITRO-,
17	0.812065	epavpst.lib	ACETONITRILE
18	0.81486	Ncptt.lib	IMP00245 Magnesite, Frankenstein, Sidesia, Chester Co., #369
19	0.815618	epavpst.lib	BENZENE, 1-CHLORO-2-NITRO-,
20	0.815643	epavpst.lib	PHENOL, 3,4-DINITRO-,

Search Options Used

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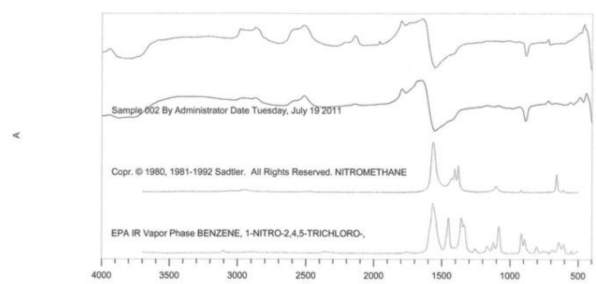
Roundup®, limestone sample

HERB_RL2.SPC Sample 002 By Administrator Date Tuesday, March 15 2011



Hit #	Quality	Library	Memo
1	**0.139644**	**Ncptt.lib**	**Sample 002 By Administrator Date Tuesday, July 19 2011**
2	**0.742437**	**srldemo.lib**	**NITROMETHANE**
3	**0.74665**	**epavpst.lib**	**BENZENE, 1-NITRO-2,4,5-TRICHLORO-,**
4	0.762443	epavpst.lib	O-CRESOL, 3-NITRO-,
5	0.764372	epavpst.lib	BENZENE, 1,4-DICHLORO-2-NITRO-,
6	0.76855	epavpst.lib	ANILINE, 4-CHLORO-N-METHYL-3- NITRO-,
7	0.769843	epavpst.lib	QUINALDINE, 8-NITRO-,
8	0.77095	epavpst.lib	OXADIAZOLE, 1,3,4-, 2-(M-FLUORO- PHENYL)-,
9	0.773657	epavpst.lib	P-CRESOL, 3-NITRO-,
10	0.774893	epavpst.lib	P-ANISIDINE, 3-NITRO-,
11	0.774917	epavpst.lib	BIPHENYL, 2,2PR-DINITRO-,
12	0.779118	epavpst.lib	PHENOL, 3,4-DINITRO-,
13	0.779895	epavpst.lib	BENZENE, 1-CHLORO-2-NITRO-,
14	0.78261	epavpst.lib	BENZENE, 1,2-DICHLORO-3-NITRO-,
15	0.783756	epavpst.lib	TOLUENE, 2,6-DICHLORO-3-NITRO-,
16	0.784092	epa_gcir.lib	ACETONITRILE
17	0.786502	srldemo.lib	FORMIC ACID
18	0.787699	epavpst.lib	2-Chloro-4-(2-propylamino)-6-ethylamino-s-triazine
19	0.788657	epavpst.lib	BENZENE, 1-IODO-2-NITRO-,
20	0.788662	epavpst.lib	TOLUENE, 2,6-DINITRO-,

Search Options Used

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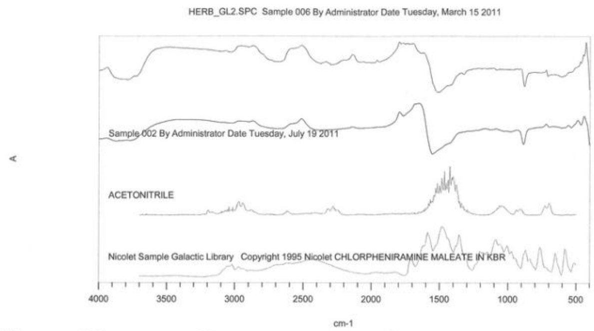
Garlon®4, limestone sample



Hit #	Quality	Library	Memo
1	**0.197477**	**Ncptt.lib**	**Sample 002 By Administrator Date Tuesday, July 19 2011**
2	**0.672986**	**epa_gcir.lib**	**ACETONITRILE**
3	**0.677344**	**nicsam.lib**	**CHLORPHENIRAMINE MALEATE IN KBR**
4	0.702989	GRAMSSuite.lib	ACETONITRILE
5	0.703335	epavpst.lib	ACETONITRILE
6	0.716395	nicsam.lib	DEXBROMPHENIRAMINE MALEATE IN KBR
7	0.745554	Ncptt.lib	IMP00025 Gray chalk, calcite with gypsum, Kremer, 5819, GCI,
8	0.751986	epavpst.lib	OXADIAZOLE, 1,3,4-, 2-(M-FLUORO- PHENYL)-,
9	0.769519	epavpst.lib	P-ANISIDINE, 3-NITRO-,
10	0.779001	epavpst.lib	PHENOL, 2,4-DICHLORO-6-NITRO-,
11	0.780628	epavpst.lib	ANILINE, 4-CHLORO-N-METHYL-3- NITRO-,
12	0.784329	epavpst.lib	STYRENE, 2,5-DIMETHOXY-B-METHYL-B- NITRO-,
13	0.784947	epavpst.lib	PYRIDINE, 2-(P-CHLOROANILINO)-,
14	0.787783	epavpst.lib	O-CRESOL, 3-NITRO-,
15	0.787901	epavpst.lib	HYDANTOIN, 5,5-DIMETHYL-2,4-DI- THIO-,
16	0.787914	epavpst.lib	BIPHENYL, 2,2PR-DINITRO-,
17	0.787922	epavpst.lib	P-CRESOL, 3-NITRO-,
18	0.789372	epavpst.lib	O-CRESOL, 5-NITRO-,
19	0.789759	epavpst.lib	BENZENE, 1-NITRO-2,4,5-TRICHLORO-,
20	0.793189	epavpst.lib	BENZENE, 1,2-DICHLORO-4,5-DI- METHOXY-,

Search Options Used

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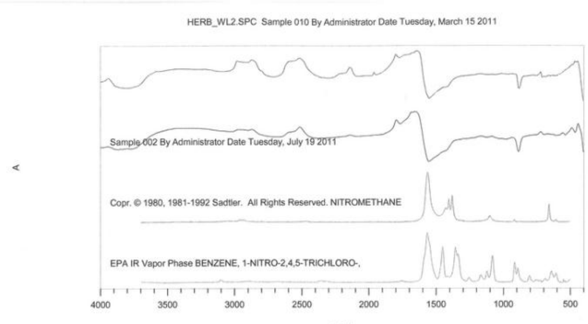
DI water, limestone sample



Hit #	Quality	Library	Memo
1	**0.145934**	**Ncptt.lib**	**Sample 002 By Administrator Date Tuesday, July 19 2011**
2	**0.757222**	**srldemo.lib**	**NITROMETHANE**
3	**0.757975**	**epavpst.lib**	**BENZENE, 1-NITRO-2,4,5-TRICHLORO-,**
4	0.765638	Ncptt.lib	IMP00025 Gray chalk, calcite with gypsum, Kremer, 5819, GCI,
5	0.771898	epavpst.lib	O-CRESOL, 3-NITRO-,
6	0.773678	epavpst.lib	BENZENE, 1,4-DICHLORO-2-NITRO-,
7	0.779001	epavpst.lib	OXADIAZOLE, 1,3,4-, 2-(M-FLUORO- PHENYL)-,
8	0.780589	epa_gcir.lib	ACETONITRILE
9	0.782472	epavpst.lib	PHENOL, 3,4-DINITRO-,
10	0.78386	epavpst.lib	P-CRESOL, 3-NITRO-,
11	0.784012	epavpst.lib	QUINALDINE, 8-NITRO-,
12	0.784082	epavpst.lib	ANILINE, 4-CHLORO-N-METHYL-3- NITRO-,
13	0.785453	epavpst.lib	BIPHENYL, 2,2PR-DINITRO-,
14	0.787249	epavpst.lib	P-ANISIDINE, 3-NITRO-,
15	0.789415	GRAMSSuite.lib	ACETONITRILE
16	0.789664	epavpst.lib	ACETONITRILE
17	0.792666	Ncptt.lib	IMP00245 Magnesite, Frankenstein, Sidesia, Chester Co., #369
18	0.792955	epavpst.lib	BENZENE, 1-CHLORO-2-NITRO-,
19	0.795119	nicsam.lib	DEXBROMPHENIRAMINE MALEATE IN KBR
20	0.795654	epavpst.lib	TOLUENE, 2,6-DICHLORO-3-NITRO-,

Search Options Used

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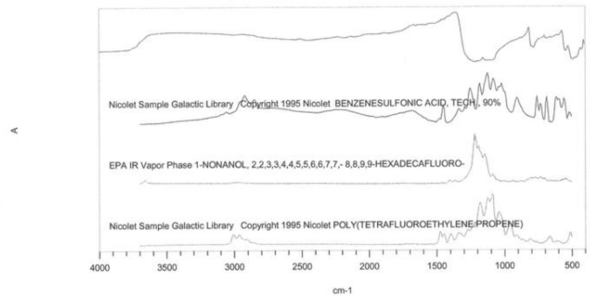
No treatment, cement sample

HERB_NC1.SPC Sample 003 By Administrator Date Thursday, March 17 2011



Hit #	Quality	Library	Memo
1	**0.614**	**nicsam.lib**	**BENZENESULFONIC ACID, TECH., 90%**
2	**0.622114**	**epavpst.lib**	**1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLU(**
3	**0.633185**	**nicsam.lib**	**POLY(TETRAFLUOROETHYLENE:PROPENE)**
4	0.65187	epavpst.lib	1,5-PENTANEDIOL, 2,2,3,3,4,4-HEXA- FLUORO-,
5	0.653098	epavpst.lib	2,2,3,3,4,4,5,5,6,6,7,7-DODECAFLUORO-1-HEPTANOL
6	0.654035	GRAMSSuite.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
7	0.654225	epa_gcir.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
8	0.669979	nicsam.lib	POLY(DIMETHYLSILOXANE)
9	0.687741	srldemo.lib	FLUOREL FC-2170*LOW VISCOSITY FLUOROELASTOMER
10	0.688682	nicsam.lib	POLY(METHYLPHENYLSILOXANE)
11	0.694476	epa_gcir.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
12	0.695336	epavpst.lib	2,2,3,3,3-PENTAFLUORO-1-PROPANOL
13	0.695664	GRAMSSuite.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
14	0.701143	epavpst.lib	2-PROPANOL, 1,1,1-TRIFLUORO-, DL-,
15	0.702305	Ncptt.lib	IMP00053 Barite, Ward's, 45E0120, MMA, tran
16	0.704265	Ncptt.lib	IMP00325 Verdigris, blue, basic, Forbes, SCC, tran
17	0.709074	epavpst.lib	NONANOIC ACID, 2,2,3,3,4,4,5,5,- 6,6,7,7,8,8,9,9-HEXADECA
18	0.713019	epa_gcir.lib	ETHANOL, 2,2,2-TRIFLUORO-,
19	0.713585	epavpst.lib	ETHANOL, 2,2,2-TRIFLUORO-,
20	0.713917	nicsam.lib	PENTAFLUOROPROPIONIC ANHYDRIDE, 99%

Search Options Used

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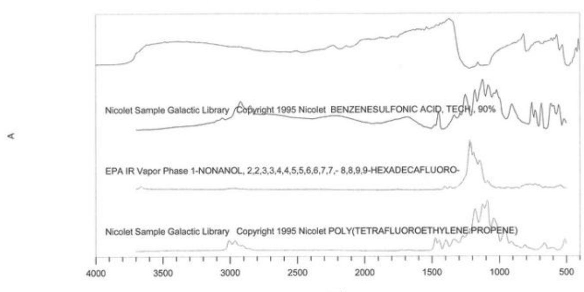
Roundup®, cement sample

HERB_RC2.SPC Sample 003 By Administrator Date Tuesday, March 15 2011



Hit #	Quality	Library	Memo
1	**0.576674**	**nicsam.lib**	**BENZENESULFONIC ACID, TECH., 90%**
2	**0.661945**	**epavpst.lib**	**1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLU(**
3	**0.673157**	**nicsam.lib**	**POLY(TETRAFLUOROETHYLENE:PROPENE)**
4	0.688099	Ncptt.lib	IMP00325 Verdigris, blue, basic, Forbes, SCC, tran
5	0.690576	epavpst.lib	2,2,3,3,4,4,5,5,6,6,7,7-DODECAFLUORO-1-HEPTANOL
6	0.691521	GRAMSSuite.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
7	0.691873	epa_gcir.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
8	0.699348	epavpst.lib	1,5-PENTANEDIOL, 2,2,3,3,4,4-HEXA- FLUORO-,
9	0.717039	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
10	0.721856	srldemo.lib	FLUOREL FC-2170*LOW VISCOSITY FLUOROELASTOMER
11	0.7223	nicsam.lib	POLY(DIMETHYLSILOXANE)
12	0.723108	Ncptt.lib	IMP00046 Verdigris, basic, synthetic, Kremer, 4445, GCI, tran
13	0.723124	epa_gcir.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
14	0.723897	epavpst.lib	2,2,3,3,3-PENTAFLUORO-1-PROPANOL
15	0.724464	GRAMSSuite.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
16	0.725753	nicsam.lib	POLY(METHYLPHENYLSILOXANE)
17	0.732274	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
18	0.73505	Ncptt.lib	IMP00191 Siderite, Ivigtut, Greenland, SI-NMNH, #132234, PM
19	0.739007	epavpst.lib	2-PROPANOL, 1,1,1-TRIFLUORO-, DL-,
20	0.741186	epavpst.lib	HEPTANE, 1-IODOPENTADECAFLUORO-,

Search Options Used

Search Type:	Spectrum
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Algorithm:	Correlation
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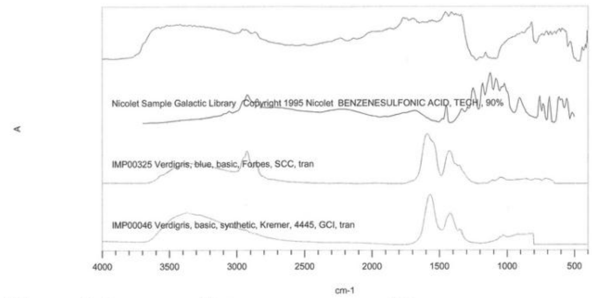
Spectrum:	C:\pel_data\Caitelin\HERB_RC2.SPC (1 of 1)

Garlon®4, cement sample

HERB_GC2.SPC Sample 007 By Administrator Date Tuesday, March 15 2011



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1	**0.6117**	**nicsam.lib**	**BENZENESULFONIC ACID, TECH., 90%**
2	**0.660118**	**Ncptt.lib**	**IMP00325 Verdigris, blue, basic, Forbes, SCC, tran**
3	**0.668455**	**Ncptt.lib**	**IMP00046 Verdigris, basic, synthetic, Kremer, 4445, GCI, tra**
4	0.710315	Ncptt.lib	IMP00264 Artinite, UD, #D-1358, PMA, tran
5	0.715789	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
6	0.724285	nicsam.lib	POLY(TETRAFLUOROETHYLENE:PROPENE)
7	0.72591	epavpst.lib	1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLUC
8	0.733822	nicsam.lib	CARBROMAL IN KBR
9	0.738601	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
10	0.742521	epa_gcir.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
11	0.744385	epavpst.lib	2,2,3,3,4,4,5,5,6,6,7,7-DODECAFLUORO-1-HEPTANOL
12	0.744741	GRAMSSuite.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
13	0.746013	epavpst.lib	1,5-PENTANEDIOL, 2,2,3,3,4,4-HEXA- FLUORO-,
14	0.751835	Ncptt.lib	IMP00289 Aurichalcite, Mapimi, Durango, MX, SI-NMNH, #1246
15	0.753766	nicsam.lib	POLY(DIMETHYLSILOXANE)
16	0.761009	nicsam.lib	POLY(METHYLPHENYLSILOXANE)
17	0.763971	Ncptt.lib	IMP00152 Verdigris, neutral, Forbes: Buckner, PMA# K-52, PM/
18	0.764435	srldemo.lib	FLUOREL FC-2170*LOW VISCOSITY FLUOROELASTOMER
19	0.764747	Ncptt.lib	IMP00317 Malachite, Forbes, SCC, tran
20	0.765253	Ncptt.lib	IMP00191 Siderite, Ivigtut, Greenland, SI-NMNH, #132234, PM/

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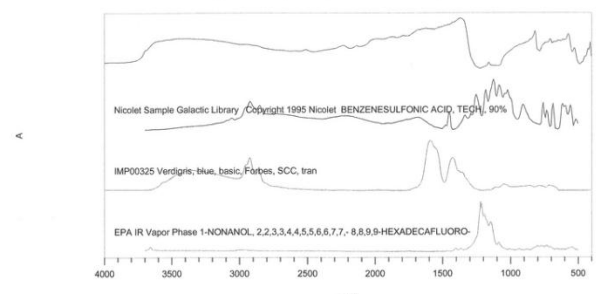
DI water, cement sample

HERB_WC2.SPC Sample 011 By Administrator Date Tuesday, March 15 2011



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1	**0.562736**	**nicsam.lib**	**BENZENESULFONIC ACID, TECH., 90%**
2	**0.673131**	**Ncptt.lib**	**IMP00325 Verdigris, blue, basic, Forbes, SCC, tran**
3	**0.674231**	**epavpst.lib**	**1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLU(**
4	0.678817	nicsam.lib	POLY(TETRAFLUOROETHYLENE:PROPENE)
5	0.700406	epavpst.lib	2,2,3,3,4,4,5,5,6,6,7,7-DODECAFLUORO-1-HEPTANOL
6	0.701176	GRAMSSuite.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
7	0.701443	epa_gcir.lib	1-HEPTANOL, 2,2,3,3,4,4,5,5,6,6,7,7- DODECAFLUORO-,
8	0.702722	Ncptt.lib	IMP00063 Chalconatronite, synthetic, Gettens, Sam #2, FGA, tr
9	0.704481	Ncptt.lib	IMP00046 Verdigris, basic, synthetic, Kremer, 4445, GCI, tran
10	0.705674	epavpst.lib	1,5-PENTANEDIOL, 2,2,3,3,4,4-HEXA- FLUORO-,
11	0.707357	Ncptt.lib	IMP00074 Malachite, Cargille, #52/M-4, WACC, tran
12	0.72068	nicsam.lib	POLY(DIMETHYLSILOXANE)
13	0.72817	epa_gcir.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
14	0.729131	epavpst.lib	2,2,3,3,3-PENTAFLUORO-1-PROPANOL
15	0.729764	GRAMSSuite.lib	1-PROPANOL, 2,2,3,3,3-PENTAFLUORO-,
16	0.73137	nicsam.lib	POLY(METHYLPHENYLSILOXANE)
17	0.732374	Ncptt.lib	IMP00191 Siderite, Ivigtut, Greenland, SI-NMNH, #132234, PM/
18	0.732524	Ncptt.lib	IMP00264 Artinite, UD, #D-1358, PMA, tran
19	0.732683	srldemo.lib	FLUOREL FC-2170*LOW VISCOSITY FLUOROELASTOMER
20	0.737069	Ncptt.lib	IMP00053 Barite, Ward's, 45E0120, MMA, tran

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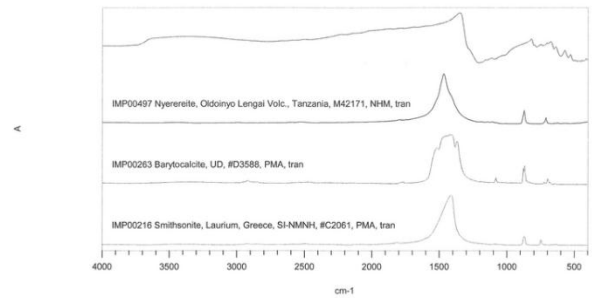
No treatment, granite sample

HERB_NG1.SPC Sample 004 By Administrator Date Thursday, March 17 2011



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1	**0.669966**	**Ncptt.lib**	**IMP00497 Nyerereite, Oldoinyo Lengai Volc., Tanzania, M42**
2	**0.678468**	**Ncptt.lib**	**IMP00263 Barytocalcite, UD, #D3588, PMA, tran**
3	**0.695199**	**Ncptt.lib**	**IMP00216 Smithsonite, Laurium, Greece, SI-NMNH, #C2061,**
4	0.696608	Ncptt.lib	IMP00287 Barytocalcite, Alston Moor, UK, SI-NMNH, #155855,
5	0.69994	Ncptt.lib	IMP00192 Calcite, Rossie, St. Lawrence Co. NY, SI-NMNH, 80
6	0.700782	Ncptt.lib	IMP00261 Vaterite, Osceola Mine, Houghton, MI, SINMNH, 169
7	0.70597	nicsam.lib	MESORIDAZINE BENZENESULPHONATE
8	0.720363	nicsam.lib	BENZENESULFONIC ACID, TECH., 90%
9	0.723116	Ncptt.lib	IMP00283 Ankerite, Eisenera, Steyer-Smark, DE, Chester, #30
10	0.724945	Ncptt.lib	IMP00214 Rhodochrosite, Catamarca, Argentina, SINMNH, #12
11	0.729877	nicsam.lib	PENICILLIN G POTASSIUM IN KBR
12	0.729952	Ncptt.lib	IMP00253 Calcite chalk, source unknown, #18264, PMA, tran
13	0.731697	Ncptt.lib	IMP00244 Siderite, Roxbury, CT, US, Chester Collection, #3088
14	0.732756	Ncptt.lib	IMP00135 Marble, paonazzo, Carrara, W. Danesi, PMA, tran
15	0.732823	Ncptt.lib	IMP00240 Calcite, Joplin, MO, US, Chester Collection, AHC301
16	0.733993	Ncptt.lib	IMP00245 Magnesite, Frankenstein, Sidesia, Chester Co., #369
17	0.73425	Ncptt.lib	IMP00136 Marble, cremino, Carrara, W. Danesi, PMA, tran
18	0.734502	nicsam.lib	SODIUM HYDROXIDE IN KBR
19	0.734759	Ncptt.lib	IMP00251 Aragonite, Bastennes, Landes, France, SINMNH, B1
20	0.738813	Ncptt.lib	IMP00174 Dolomite, Oberdorf Styria, Austria, SI-NMNH, #R125

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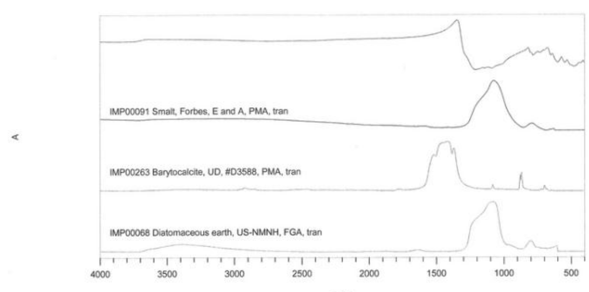
Roundup®, granite sample

HERB_RG2.SPC Sample 004 By Administrator Date Tuesday, March 15 2011



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1	**0.636578**	**Ncptt.lib**	**IMP00091 Smalt, Forbes, E and A, PMA, tran**
2	**0.667144**	**Ncptt.lib**	**IMP00263 Barytocalcite, UD, #D3588, PMA, tran**
3	**0.669115**	**Ncptt.lib**	**IMP00068 Diatomaceous earth, US-NMNH, FGA, tran**
4	0.67788	Ncptt.lib	IMP00497 Nyerereite, Oldoinyo Lengai Volc., Tanzania, M42171
5	0.682956	Ncptt.lib	IMP00287 Barytocalcite, Alston Moor, UK, SI-NMNH, #155855,
6	0.691054	Ncptt.lib	IMP00216 Smithsonite, Laurium, Greece, SI-NMNH, #C2061, P
7	0.69357	Ncptt.lib	IMP00192 Calcite, Rossie, St. Lawrence Co. NY, SI-NMNH, 80
8	0.703356	Ncptt.lib	IMP00261 Vaterite, Osceola Mine, Houghton, MI, SINMNH, 169
9	0.7036	Ncptt.lib	IMP00233 Cristobalite, Glass Mt, Coso R., CA, SI-NMNH, 1365
10	0.705691	Ncptt.lib	IMP00426 Egyptian green, Doerner-Institut Collection, DI, tran
11	0.706737	epavpst.lib	1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLU
12	0.710483	Ncptt.lib	IMP00214 Rhodochrosite, Catamarca, Argentina, SINMNH, #12
13	0.711782	nicsam.lib	MESORIDAZINE BENZENESULPHONATE
14	0.714196	Ncptt.lib	IMP00361 Green earth (Terra verde antica), wet KBr, Zecchi, U
15	0.716664	nicsam.lib	POLY(TETRAFLUOROETHYLENE:PROPENE)
16	0.716987	Ncptt.lib	IMP00127 Flake white, all basic lead carb, Forbes: Roberson, P
17	0.718421	Ncptt.lib	IMP00107 Lead white, all basic lead carbonate, source unknow
18	0.718698	Ncptt.lib	IMP00283 Ankerite, Eisenera, Steyer-Smark, DE, Chester, #30
19	0.718839	epavpst.lib	1,5-PENTANEDIOL, 2,2,3,3,4,4-HEXA- FLUORO-,
20	0.721396	Ncptt.lib	IMP00053 Barite, Ward's, 45E0120, MMA, tran

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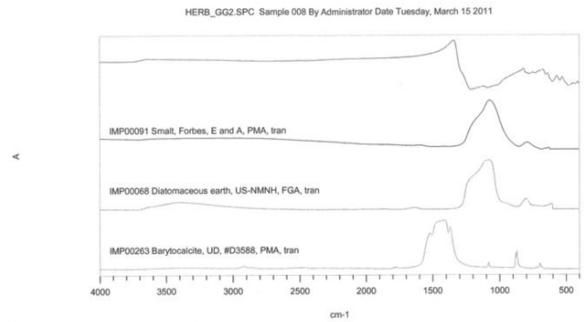
Garlon®4, granite sample



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2	**0.667701**	**Ncptt.lib**	**IMP00068 Diatomaceous earth, US-NMNH, FGA, tran**
3	**0.66979**	**Ncptt.lib**	**IMP00263 Barytocalcite, UD, #D3588, PMA, tran**
4	0.675659	Ncptt.lib	IMP00497 Nyerereite, Oldoinyo Lengai Volc., Tanzania, M42171
5	0.680828	Ncptt.lib	IMP00426 Egyptian green, Doerner-Institut Collection, DI, tran
6	0.686226	Ncptt.lib	IMP00287 Barytocalcite, Alston Moor, UK, SI-NMNH, #155855,
7	0.687308	Ncptt.lib	IMP00233 Cristobalite, Glass Mt, Coso R., CA, SI-NMNH, 1365
8	0.69164	Ncptt.lib	IMP00216 Smithsonite, Laurium, Greece, SI-NMNH, #C2061, P
9	0.694423	Ncptt.lib	IMP00192 Calcite, Rossie, St. Lawrence Co. NY, SI-NMNH, 808
10	0.697356	Ncptt.lib	IMP00361 Green earth (Terra verde antica), wet KBr, Zecchi, UI
11	0.706157	Ncptt.lib	IMP00261 Vaterite, Osceola Mine, Houghton, MI, SINMNH, 169
12	0.707107	nicsam.lib	MESORIDAZINE BENZENESULPHONATE
13	0.711331	Ncptt.lib	IMP00214 Rhodochrosite, Catamarca, Argentina, SINMNH, #12
14	0.716919	Ncptt.lib	IMP00037 Ochre, yellow, burnt, Kremer-Pigmente, 4033, GCI, t
15	0.717208	epavpst.lib	1-NONANOL, 2,2,3,3,4,4,5,5,6,6,7,7,- 8,8,9,9-HEXADECAFLUC
16	0.718479	Ncptt.lib	IMP00422 Umber, raw with goethite, silicate, Dolci & Figli, CCI,
17	0.718635	nicsam.lib	PSILOCYBIN IN KBR
18	0.719417	nicsam.lib	POLY(TETRAFLUOROETHYLENE:PROPENE)
19	0.719827	Ncptt.lib	IMP00283 Ankerite, Eisenera, Steyer-Smark, DE, Chester, #30
20	0.722356	Ncptt.lib	IMP00102 Smalt, No. 1, Forbes: Newman, PMA# J-65, PMA, tr

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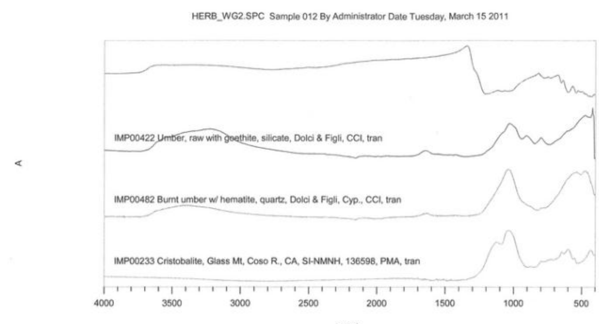
DI water, granite sample



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2	**0.685705**	**Ncptt.lib**	**IMP00482 Burnt umber w/ hematite, quartz, Dolci & Figli, Cy**
3	**0.688466**	**Ncptt.lib**	**IMP00233 Cristobalite, Glass Mt, Coso R., CA, SI-NMNH, 13**
4	0.688609	nicsam.lib	MESORIDAZINE BENZENESULPHONATE
5	0.68892	Ncptt.lib	IMP00497 Nyerereite, Oldoinyo Lengai Volc., Tanzania, M42171
6	0.704651	Ncptt.lib	IMP00389 Umber, burnt, hematite with silicate clay, Zecchi, UI,
7	0.704686	Ncptt.lib	IMP00365 Terra rossa w/ kaolinite, hematite, Dolci & Figli, CCI,
8	0.705131	Ncptt.lib	IMP00263 Barytocalcite, UD, #D3588, PMA, tran
9	0.707612	Ncptt.lib	IMP00458 Sienna, burnt, w/ hematite, kaolinite, Dolci & Figli, CC
10	0.714209	Ncptt.lib	IMP00361 Green earth (Terra verde antica), wet KBr, Zecchi, UI
11	0.719222	Ncptt.lib	IMP00216 Smithsonite, Laurium, Greece, SI-NMNH, #C2061, P
12	0.723589	Ncptt.lib	IMP00192 Calcite, Rossie, St. Lawrence Co. NY, SI-NMNH, 808
13	0.725117	Ncptt.lib	IMP00287 Barytocalcite, Alston Moor, UK, SI-NMNH, #155855,
14	0.72606	Ncptt.lib	IMP00426 Egyptian green, Doerner-Institut Collection, DI, tran
15	0.726312	nicsam.lib	PENICILLIN G POTASSIUM IN KBR
16	0.726641	nicsam.lib	SODIUM HYDROXIDE IN KBR
17	0.727385	nicsam.lib	3C2\LANTHANUM SULFATE NONAHYDRATE, 99.999%, GOL
18	0.727787	Ncptt.lib	IMP00261 Vaterite, Osceola Mine, Houghton, MI, SINMNH, 169
19	0.732742	nicsam.lib	BENZENESULFONIC ACID, TECH., 90%
20	0.744133	Ncptt.lib	IMP00091 Smalt, Forbes, E and A, PMA, tran

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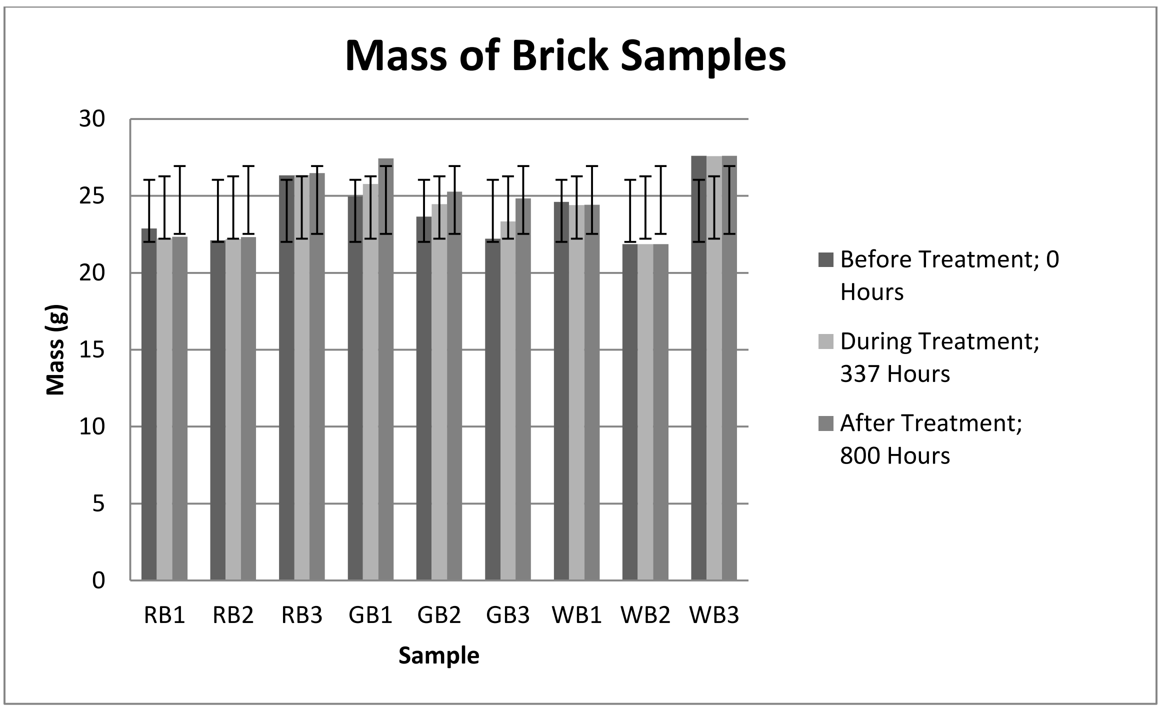
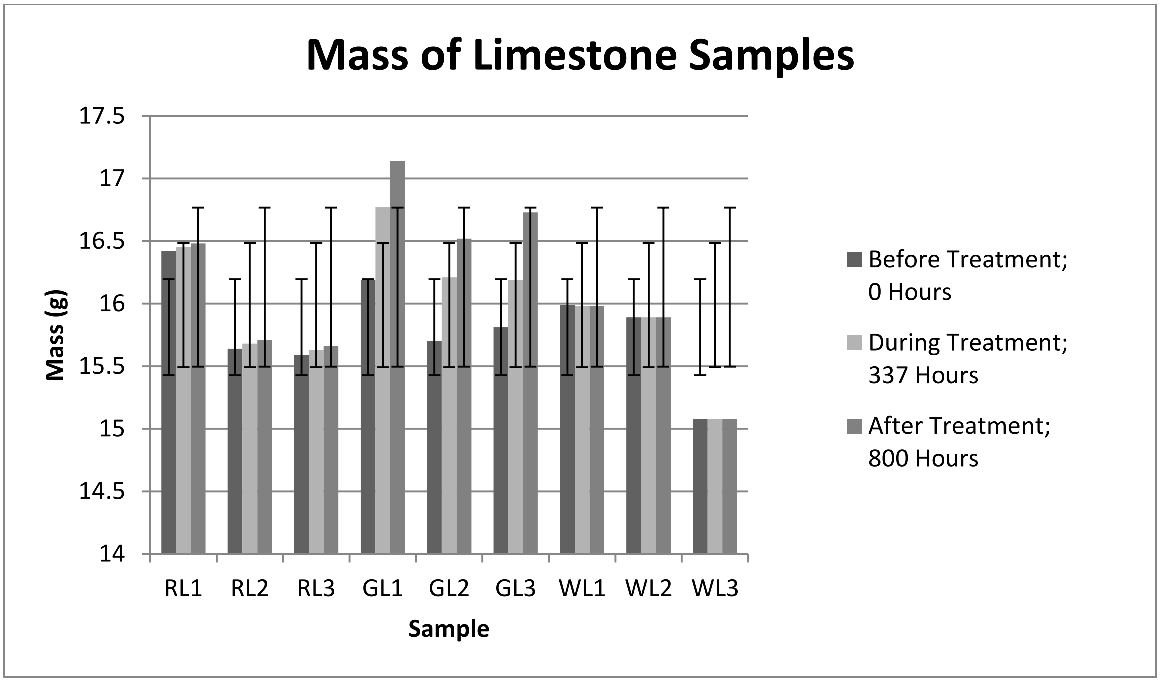
APPENDIX H

MASS DATA

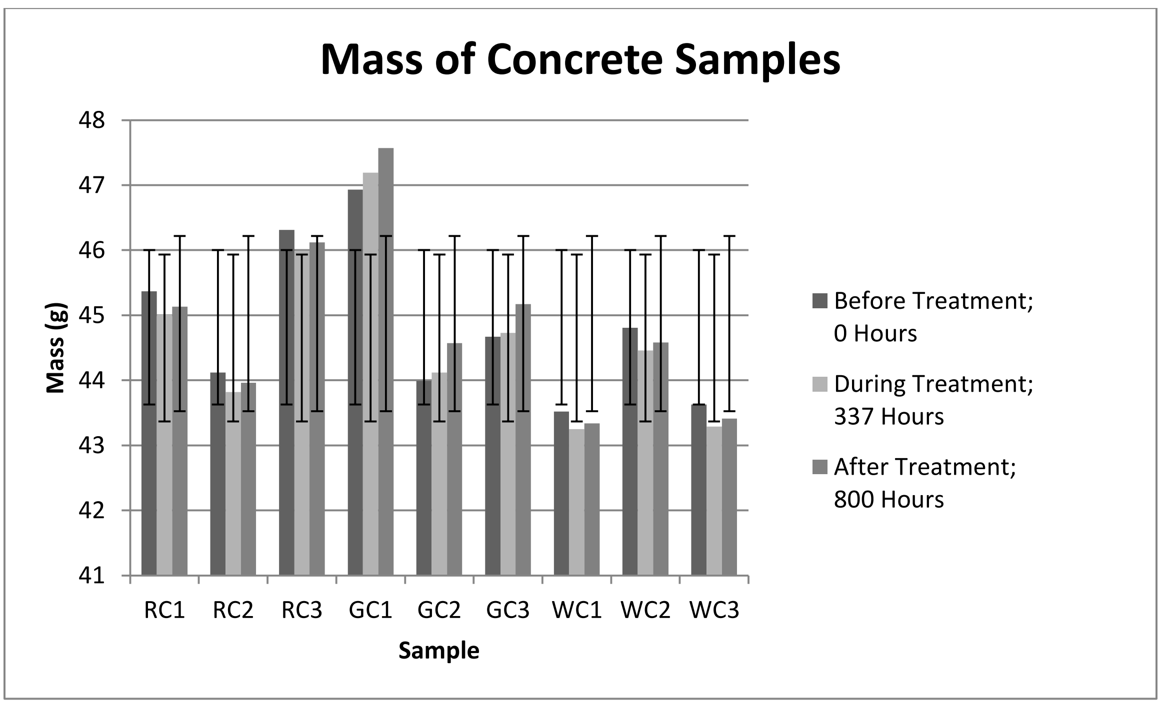
The following figures represent the changes in mass of each of the sample throughout the experiment. Error bars represent the standard deviation.



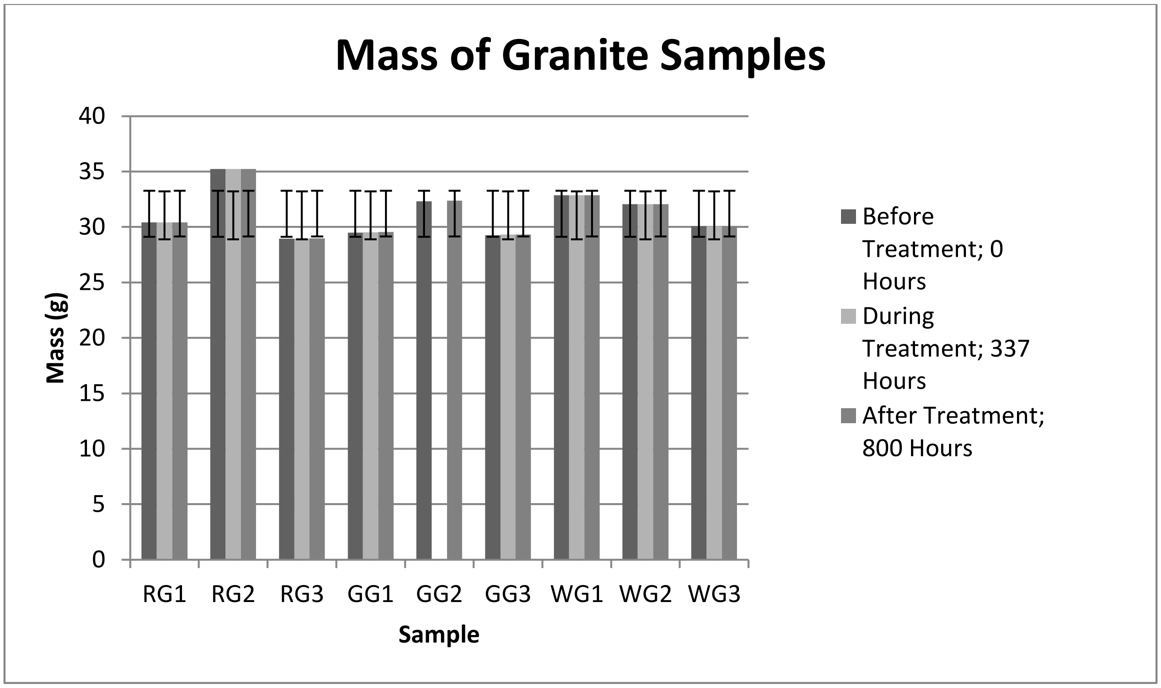
The change in the mass of the brick samples throughout the experiment.



The change in the mass of the limestone samples throughout the experiment.



The change in the mass of the concrete samples throughout the experiment.



The change in the mass of the granite samples throughout the experiment.