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EVALUATION OF HYGROSCOPIC SOIL AMENDMENTS AND NATURAL FREEZE-THAW CYCLING TO ACCELERATE THE MECHANICAL BREAKDOWN OF ARTIFACTS IN DEMOLITION SITE SOILS

by

PHILLIP J. BACKERS

THESIS

Submitted to the Graduate School

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for the degree of

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Approved By:

Advisor

Date

DEDICATION

TO MY WIFE, KELLY, WHO HAS HELPED AND GUIDED ME THROUGH GRADUATE SCHOOL IN MORE WAYS THAN I COULD POSSIBLY LIST HERE.

ACKNOWLEDGMENTS

TO MY FRIEND AND MENTOR, DR. HOWARD. YOU PROVIDED ME WITH ENDLESS GUIDANCE, KNOWLEDGE, AND SUPPORT DURING THE COURSE OF THIS STUDY.

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1 Introduction

In many urban areas, building demolition has created large areas of vacant land. This open space has attracted considerable attention as a potential resource. Urban gardening has already become a popular way of repurposing vacant land created by urban renewal. There is also great interest in using vacant land for urban farming on a much larger scale. The problem is that rock-like artifacts (objects of anthropogenic origin), particularly brick, concrete, and mortar, are often very abundant in demolition site soils. Excessive artifact content and soil compaction can severely restrict plant growth, microbial activity, and the infiltration of water (Soil Survey Staff, 2010). The USEPA (2011ab) has recommended physically removing artifacts as a way of preparing the site for future use. The problem is that it is not practical or economically feasible to remove these materials on a scale large enough for urban farming.

Due to urban decay and large blighted areas, Detroit is one city now faced with the problem of revitalizing large areas of vacant land. The Detroit Land Bank Authority currently possesses about 90,000 vacant properties in Detroit (Gallagher, 2015). On average, the City of Detroit demolishes 5,000 vacant structures each year (SEMCOG, 2010). Since most of the vacant land in the City of Detroit is comprised of urban soils, finding a way to revive these areas is increasingly becoming a top priority. A method for generating reclaimed soils from demolition sites is needed in order to deal with the growing problem of unusable urban land. Reclaimed soils would allow for increased permeability and would also facilitate plant growth.

As an alternative to physical removal, the purpose of this study was to investigate the feasibility of using hygroscopic materials as soil amendments to promote the physical

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breakdown of rock-like artifacts *in situ* using natural freeze-thaw/wet-dry cycles. Hygroscopic materials are compounds which possess an ability to attract and hold on to water molecules. Hygroscopic materials are well known to promote the physical deterioration of construction materials (e.g., concrete highway structures) via natural freeze-thaw cycles. Freeze-thaw cycling has also been shown to have strong effects on the physical properties of soil, and the microbial communities present within the soil (Henry, 2007). The purpose of this study was to test the hypothesis that the effects of freeze-thaw cycling, combined with a hygroscopic amendment, will accelerate the mechanical breakdown of artifacts in demolition soils and generate a reclaimed soil better suited for plant growth and urban agriculture.

2 Study Area and Geologic Setting

This study focuses on the Metro Detroit Region of Southeastern Michigan. The experiments performed during the course of this study aimed to replicated conditions in the southeastern portion of Michigan, in particular the metropolitan Detroit area. The oldest part of the city is the downtown district where the city was founded by Antoine de la Mothe Cadillac in 1701 (Burton, 1922). Buildings in this area of the city generally consist of brick, mortar, plaster, and iron which were the most common building materials during the industrial boom of the late 19th century (Dubay, 2012). Detroit underwent another industrial period during the 1920's which had a significant impact on the nature of urban soils due to recently invented power earth-moving equipment. Most recent impacts on urban soils include acid rain and the intense use of deicing salts (Dubay, 2012).

The entire area of Detroit is situated upon layers of Paleozoic sedimentary bedrock which is in turn overlain by layers of Pleistocene glacial sediments which range from 10-30 meters in thickness (Mozola, 1969). Detroit is located upon the southeastern edge of the Detroit Moraine (Howard 2010) on sediments deposited in the paleolakes of Grassmere, Elkton, and Rouge (Bay, 1938; Dubay, 2012). The city's elevation ranges from approximately 193-203 meters (Howard, 2010). The sediments in this area of Michigan typically consist of a capping of sand 2-3 m in thickness overlying weakly stratified layers of clayey diamicton with pebbles and cobbles throughout the clay matrix (Howard, 2010). The sand capping is noticeably thinner in the area of downtown Detroit, measuring 0-20 cm in thickness (Howard, 2010).

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3 Previous Work

Demolition site soils are often highly variable, containing large amounts of construction debris including concrete, brick, mortar, wood, metal, plastic, drywall, and miscellaneous trash. Figures 1 and 2 are photos of a typical vacant lot in the City of Detroit after demolition. Figures 3 and 4 show a former industrial site with excessive waste building materials. Some vacant lots have been turned into urban gardens with the raised garden approach being the most common (Fig. 5). Figure 6 depicts a simplified sequence of events when a house is demolished and turned into vacant land. These soils are also often highly compacted which causes poor drainage and aeration. A recent study by Howard and Olszewska (2011) on urban soils in the City of Detroit found that the weathering rate of artifacts increases with decreasing aggregate size, and that there is a stability sequence of artifacts: brick > steel > concrete > mortar (Dubay, 2012). Brick and concrete are composite materials (anisotropic), which is why cyclic stresses, such as freeze-thaw cycling, can cause structural failure of the material. Failure of the material can occur in either the microscopic or macroscopic scale. Brick samples were selected

for this study since they were shown to be the most resistant to weathering within an urban soil sequence.



Figure 1: Typical open-hole vacant lot after demolition.



Figure 2: A typical vacant lot after backfilling.



Figure 3: Typical vacant lot at former industrial site produced by building demolition. Extensive waste building materials have been incorporated into the anthropogenic soil. Staff is 1.2 m in height.



Figure 4: In the past, some contractors prepared a demolition site for future building construction by intentionally leaving a large volume of rock-like artifacts to form what they called "hardcore." Staff is marked in 10 cm intervals.



Figure 5: The "raised garden" approach is used by most urban gardeners in Detroit, but in this case an artifact rich site soil is being used to grow lettuce and string beans.



Fig. 9. Genetic model showing how construction and demolition processes affect demolition site stratigraphy and anthrosequence development: A, Residential site prior to construction; B, Inverted stratigraphy resulting from excavation for basement; C, Stratigraphy following demolition and backfilling. D, E, and F are map views of A, B and C, respectively. NS, native soil; CF, construction fill; DF, demolition fill; Sx, glaciolacustrine sand capping; Dc, clayey diamicton.

Most previous work pertaining to freeze-thaw cycling and its effects on soil to date has primarily focused on the qualitative effects on the soil and its structure. Ferrick and Gatto (2005) demonstrated that the rate and quantity of soil eroded due to a freeze-thaw cycle increases dramatically with increasing soil moisture content. These results were observed after only one simulated freeze-thaw cycle. This study highlights the significant effects that minimal numbers of freeze-thaw cycles can have on soil sequences. Oztas and Fayetorbay (2003) were also able to demonstrate the effects of a small number of cycles on soil. The authors studied the effects of freeze-thaw cycles on the wet aggregate stability of a soil. Different parent materials were used for the study and exposed to various numbers of cycles (3, 6, or 9) at various temperatures. Successive cycles were shown to generally decrease aggregate stability regardless of soil type or freezing temperature. Few studies have been aimed at quantifying the degrading effects freeze-

Figure 6: Simplified model showing how a demolition site soil is generated, taken from Howard et al. 2015.

thaw cycling has on artifacts and building debris buried in soil sequences. Although accurately replicating winter conditions in southeast Michigan was beyond the scope of this study, attempts have been made to correlate laboratory freeze-thaw tests with actual field conditions (Lienhart, 1988). Additionally, the number of freeze-thaw cycles completed in many previous studies have been minimal, with the most common being between one and five cycles (Henry, 2007).

Several issues must be taken into consideration when looking at the effects of seasonal freeze-thaw cycling. It is important to understand the geothermal profile of the soil to accurately predict the number and intensity of the freeze-thaw cycles that occur annually for a given geographic area. This geothermal profile "is a function of the net transfer of heat between the ground surface and the air, and the thermal properties of the subsurface soil" (Yong et al., 2012). Yong et al. (2012) identifies the main heat transfer mechanisms as radiation, convection, and conduction. Each of these mechanisms has different factors that involve the nature of the heat transfer. Figure 7 depicts the air temperature profile. This profile is a way of showing the temperature at the soil ground surface at any time (Yong et al., 2012).





Figure 7: Idealized annual air temperature profile, after Yong et al. 2012.

The geothermal profile below (Fig. 8) is taken from Yong et al. 2012 and shows the soil zone, known as the "active layer", which is susceptible to freeze-thaw cycling during the winter season. The main factor responsible for characterizing this "active layer" is how far the frost front penetrates into the soil subsurface (Yong et al., 2012). It becomes evident from this figure that a barren ground surface (free of vegetation, snow cover, etc.) is the most susceptible to frost penetration.





Figure 8: Idealized geothermal profile, taken from Yong et al. 2012.

A key aspect of this study is to understand the nature and characteristics of soil freezing and freeze-thaw cycling in southeastern Lower Michigan. Understanding the nature of freeze-thaw activity in southeastern Michigan will allow for an accurate estimate as to how many years of freeze-thaw cycling were simulated during the course of the experiment. Isard and Schaetzl (1998) studied the climatic and soil-temperature data from five winter seasons in southern Michigan in order to determine differences in soil freezing and freeze-thaw cycles at a depth of 5 cm. The authors used National Weather Service data from 1951 – 1980 and looked at minimum and maximum daily temperatures and precipitation data. The data came from 56 weather stations across the Lower Peninsula. The data was analyzed using a computer algorithm which generated a temperature profile for "coarse-textured, well-drained, forested soils." An important finding

of this study was the fact that early season snowfall did not appear to be a significant inhibitor to soil freezing. Isard and Schaetzl (1998) offered two explanations to support this finding; "(1) deep snowpacks usually experience at least one episode of melt, and the cold meltwater pulse from that melt event probably cools soils down faster than might an early outbreak of cold air; and (2) the coldest air temperatures occur in mid-winter, such that snowpack status at that time may be most important in the determination of whether soil freezes." The authors further suggested that mid-winter melt events may be most crucial in determining the extent of soil freezing because these events leave the soil surface uncovered and thus highly susceptible to freezing. It is then suggested that "for these reasons, southeastern Lower Michigan and the Saginaw Bay Lowlands have the highest maximum number of days with continuous subzero temperatures and the highest numbers of freeze-thaw cycles." The graph (Figure 9) below shows the areas of maximal and minimal soil freezing and is modified from Isard and Schaetzl (1998) to show the location of Detroit, Michigan. This figure shows that Detroit and the surrounding metro region are located within the areas of Michigan that experience the most frequent and intense soil freezing. The authors of the study conclude that on average the southeastern portion of the Lower Peninsula will experience 3 – 5 freeze-thaw cycles per year (Isard and Schaetzl (1998).



Figure 9: Graph showing areas of maximum soil freezing, modified from Isard and Schaetzl (1998).

Extensive work exists documenting the effects of freeze-thaw cycling on soil, concrete, and other building materials. Many studies have focused on characterizing the deterioration of concrete under freeze-thaw conditions in an effort to increase durability and service life of concrete structures (Niu et al., 2013). In contrast, the goal of this study was to identify hygroscopic substances that accelerate the breakdown of building materials like concrete. Freeze-thaw cycling and salt crystallization have been shown to be the two major causes of brick deterioration (Koroth et al., 1998). Frost action is the

driving force that causes damage to concrete and masonry structures. Frost damage occurs when moisture collects and freezes within the pore space of the material. Adsorbed water is drawn into the material mass through capillary forces (Lienhart 1988). The moisture is held in both microscopic and macroscopic pore spaces within the material. As the water freezes it expands, producing internal pressures in the material. The intensity of this internal pressure depends upon a number of factors including porosity, degree of saturation, and external temperature (Koroth et al., 1998). The term porosity describes both the amount and type of pore space within the sample. Accelerated weathering experiments have shown that the pressures induced in samples due to salt re-crystallization cause significant damage and loss of sample strength (Akin et al. 2011).

Lienhart (1988) cites work by Fukuda (1983) about pore water pressure in rocks during freezing and discusses the significance of moisture with regards to freeze-thaw cycling. Moisture content within the material has great significance and accelerates the fracturing process. Lienhart (1988) describes freezing and thawing without the presence of moisture as "merely thermal cycling." Although it is possible to fracture and degrade the samples without moisture, the presence of water greatly accelerates this process. The breakdown of material under freeze-thaw conditions is due to the fact that hydrostatic pressures are developed during the freezing process. Stresses due to hydrostatic pressures can range from approximately 2,000 psi (13.8 Mpa) to 30,000 psi (206.8 Mpa) (Powers, 1965; Bowles, 1982; Ollier, 1984; Lienhart, 1988). Figure 10 is taken from Lienhart 1988 and shows the hydrostatic pressure developed at various temperatures. For partially saturated samples, the freezing process within the material begins at the

surface, as water in the interior migrates towards the exterior, towards the freezing front. As freezing continues, the freezing front continues moving inward causing a reversal of internal pore pressures, eventually leading to hydrostatic fracturing. In fully saturated samples there is a continued increase in pore pressures leading to fracturing. Lienhart (1988) mentions several types of deterioration in rock that are likely related to freeze-thaw cycling. Partially saturated rocks are susceptible to spalling, which occurs when pore pressures build up just ahead of the advancing freezing front. The fracture plane occurs parallel to the freezing front. Splitting is another type of deterioration, more commonly occurring in rocks that are full saturated and that are anisotropic with respect to permeability. Pore pressure increases in the directions of greatest permeability causing splitting in the direction normal to the axis of greatest permeability.



Figure 10: Hydrostatic pressure versus temperature in a closed system, taken from Lienhart 1988

Freeze-thaw cycling and intensity has been studied in many parts of the world. Lienhart (1988) claimed that "the intensity of a freezing and thawing environment depends on the freezing temperature, the duration of the freezing cycle, the available moisture, the slope direction, degree of saturation, and permeability." Lienhart (1988) concludes that the intensity of the freezing and thawing cycles is thus largely dependent upon the geographic area and secondly upon the rock mass properties. Lienhart (1988) developed a "moist freeze-thaw index" in an effort to determine actual field conditions during freezethaw cycling. Figure 11 is the isoline map of the "moist freeze-thaw index" of the United States developed by Lienhart (1988). It suggests that Michigan is among states with the highest moist freeze-thaw index indicating intense freeze-thaw cycling activity.



Figure 11: Moist freeze-thaw index isoline map from Lienhart 1988.

Studies have also looked at the effects of freeze-thaw activity on aggregate stability (Oztas and Fayertorbay, 2003) and soil erosion (Ferrick and Gatto, 2005). Through their wet-sieving experiments, Oztas and Fayertorbay (2003) show that wet aggregate soil stability decreased for all soils that were studied. This is important for this study, because it suggests that reclaimed demolitions site soils may need to be protected from erosion in order to preserve the soil structure that forms. Ferrick and Gatto (2005) showed that freeze-thaw activity is a significant factor in upland soil erosion and that "FT effects can increase up to several hundred per cent with soil moisture." This result is significant factor with increased soil moisture content.

The goal of this study was to promote the breakdown of artifacts on a timescale suitable for remediation activities in the near future, using amendments which are benign in terms of their environmental impact. The results of previous work suggest that a 3% solution should be used for amendment concentrations, and given 3 to 5 freeze thaw cycles per year in Detroit, a total of 24 cycles will simulate 5 to 8 years of weathering (Hudec, 1999; Isard and Schaetzl, 1998). The use of fertilizer salts as amendments would be ideal because they would not only promote the physical breakdown of artifacts, but also enhance soil fertility. Given that excess salt content is well known to adversely affect plant growth, the pH and electrical conductivity of the soils in each bin were measured after completion of the freeze-thaw/wet-dry experiment.

4 Materials and Methods

The following paragraphs outline the various materials and methods used during the course of this investigation. Experimental set-ups included simulated freeze-thaw cycling, wet-dry cycling, and compression strength testing.

4.1 Brick Samples and Soil

The freeze-thaw cycling experiment was carried out using 10 plastic bins measuring 43 x 30 x 18 cm that were filled with commercially available topsoil. The topsoil was characterized as a black silty loam with trace amounts of gravel and organics. This type of topsoil was used because it is easy to obtain in large quantities and would be a consistent soil type for each of the bins. The samples used in the study were reclaimed bricks obtained from a local building supply store. Each of the different types of brick was cut into rectangular pieces measuring approximately 50 x 50 x 100 cm. Cutting the brick samples into rectangular prisms yielded a height to thickness ratio (h/t) of approximately 2 (Fig. 12). The general rule for h/t ratio is between 1.3 and 5 for compression strength testing. The brick samples consisted of clay, concrete, and sand-lime. The table below describes the different brick types used in this study. Sand-Lime brick was used to simulate mortar artifacts which are especially common in urban soils. Table 1 is a summary of the brick samples used in the study.



Figure 12: Cut brick samples used for the simulated weathering experiment.

Table 1	: Brick	samples	and	types.
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Brick	Name (Abbreviation)	Туре
1	Daniel (D)	Clay
2	Belden (IB)	Clay
3	Western Brick Co., Danville, Illinois (W)	Clay
4	Banfield (B)	Clay
5	Unidentified (U)	Clay
6	Belden, Canton, Ohio (IIB)	Clay
7	Jefco (J)	Sand-Lime
8	Concrete (C)	Concrete

4.2 Freeze-Thaw Cycling Experiment

The cut brick pieces were buried in the middle portion of the plastic bin in order to simulate waste building materials buried in a demolition site soil. Each soil bin was then treated with a different hygroscopic amendment (Table 2). Figure 13 shows the bins with brick samples prior to being filled with soil. The bins were then filled with a layer of soil, as seen in Figure 14.

Bin No.	Amendment
1	Water
2	Sodium Chloride
3	Calcium Chloride
4	Magnesium Sulfate
5	Ammonium Sulfate
6	Aluminum Sulfate
7	Urea
8	Salt Mix
9	Sugar
10	Humic Acid

Table 2: Soil amendments used for freeze-thaw cycling experiment.



Figure 13: Ten plastic bins with brick samples partially buried in topsoil.

Each amendment was weighed out to 60 grams and dissolved in 2 liters of deionized water. The 2 liters of 3% (by weight) solution was applied to each bin before the start of freeze-thaw cycling. The initial application was the only time each amendment was applied to the soil bins. Each complete freeze-thaw cycle consisted of a 24-hour freeze at approximately -23° C followed by a 48-hour thaw period at approximately 20° C. The thawing period increases the volume of the pore space in the brick, allowing the liquid water to flow further into the pore space and micro-cracks in the sample (Yu et al., 2015). The repeated freezing and thawing further increases the abundance of microcracks in the sample, causing a significant reduction in compressive strength and resistance to further thermal cycling episodes (Nicholson et al., 2000). The goal of the cycling period was not to replicate real world circumstances, but rather to ensure a complete freeze and thaw of

the simulated soil column. However, it could be argued that the freeze-thaw cycle method discussed above does simulate large diurnal temperature fluctuations during the spring season. During these large diurnal cycles, the soil column can experience daily temperature fluctuations of greater than 20°C (Henry, 2007). The temperature of the soil column was checked regularly during each freeze-thaw cycle with a probe thermometer to ensure a complete freeze was achieved. The soil bins were not disturbed at any time during the cycling experiment. A total of 24 cycles was completed, simulating approximately 5 to 8 years of natural freeze-thaw activity (Isard and Schaetzl, 1998).



Figure 14: Example showing arrangement of brick samples in simulated soil.

4.3 Wet-Dry Cycling Experiment

Following the completion of the freeze-thaw cycling experiment, eight of the most resilient brick samples were set aside for additional testing. The additional testing
consisted of repeated wetting and drying of the samples with an amendment in a simulated fill soil (Fig. 15 and 16). The samples used for the wet-dry testing were four concrete brick samples and four red clay brick samples. The experiment was carried out in four aluminum bread pans. Two samples were buried with commercially available topsoil in each pan. Two of the pans were treated with an initial application of 1 liter of a 3% (by weight) solution of the salt mixture. The remaining two pans were treated with 1 liter of a 3% (by weight) solution of sugar.



Figure 15: Wet-Dry experimental set up (salt solution).



Figure 16: Wet-Dry experimental set up (sugar solution)

The pans were allowed to soak for a period of 24 hours at room temperature (20° C) before being oven-dried for 24 hours at a temperature of 90° C. This process constituted one complete wet-dry cycle. The experiment ran for a total of 20 complete cycles. After the initial amendment application, the pans were re-wetted with deionized water for the next three cycles. Following the three water treatments, the pans were again given an amendment treatment. This process was repeated so that each pan received a total of four amendment treatments during the course of the experiment. Brick samples were then removed from the bins and subjected to compressive strength testing.

4.4 Compression Strength Testing

Uniaxial compression strength testing methods were used in order to quantify the degradation of the brick samples during the course of the experiments. Multiple samples of each brick type were tested prior to experimental testing in order to establish control compressive strength values. Average compressive strength was computed by averaging four tests from each brick type. Brick samples were subjected to uniaxial compression testing at the conclusion of the experiments and compared to the control values (Fig. 17 and 18). Any samples that broke apart completely during the course of the experiment or that were degraded so severely that compressive testing was unable to be conducted were assumed to have lost 100% of their compressive strength (for the purposes of statistical analysis). The brick samples were tested using a MTS Model# 660.23A-02/292.225 compression machine with an ultimate load capacity of 550 kips (~3,800 MPa). The machine determined the ultimate load at which failure of the sample occurred. The ultimate load capacity was then divided by the surface area of each sample in order to determine a compressive strength value.

Preliminary statistical analysis showed that the uniaxial compression data followed a normal distribution, hence Student's *t*-test was used to test the statistical significance of variations in brick strength using standard methods (Davis, 1986). Hence, given a mean (X) and standard deviation (S) for *n* measurements, the hypothesis tested was that $X_1 = X_2$, where

$$t = X_1 - X_2 / Se$$
,
Se = Sp $\sqrt{(1/n_1 + 1/n_2)}$, and
Sp² = [(n₁-1)S₁² + (n₂-1)S₂²]/n₁ + n₂ - 2.

If the calculated value of *t* exceeded the table value, the null hypothesis was rejected, and it was concluded that there was a statistically significant difference between means at a level of $\alpha/2$ for $n_1 + n_2 - 2$ degrees of freedom.



Figure 17: Brick sample that has undergone uniaxial compression testing.



Figure 18: Samples after compressive strength testing.

4.5 Soil Characterization

Standard methods were used to determine the pH (3:1 soil solution) and electrical conductivity of the soil. Control values were determined by measuring the pH and electrical conductivity of the soil prior to beginning freeze-thaw cycling and before the addition of any soil amendment. Soil from each bin was allowed to dry and weighed out to 10 gram samples upon conclusion of the freeze-thaw cycling. The samples were then combined with 30 grams of distilled water and allowed to soak for 1 hour. The soil solution of each sample was measured for both pH and electrical conductivity. Visual observations of each soil bin were recorded for both the freeze-thaw cycling and wet-dry cycling experiments.

4.6 Brick Microporosity Analysis

Thin section samples (clay and concrete brick) were prepared from control bricks and from the samples that had undergone both freeze-thaw cycling and wet-dry cycling. The samples were compared to one another and analyzed for features such as mineral crystallization within pore space, micro-cracking, and pitting of the brick surface. Microporosity was measured by point counting thin sections stained blue for void space. Point counting was done by counting 400 points per slide. The null hypothesis that proportions p₁ and p₂ were the same was tested using Student's *t*-test following the method of Dixon and Massey (1957). Thus, given two random samples of size n₁ and n₂, and the corresponding proportions p₁ = X₁/n₁ and p₂ = X₂/n₂, where X₁ and X₂ are the numbers of pores counted, the difference between p₁ and p₂ may be estimated by the following confidence interval: p₁ - p₂ ± t $\sqrt{p_1(1 - p_1)/n_1} + p_2(1-p_2)/n_2$, where *t* is the tabulated value of Student's *t*-distribution for $\alpha/2$ level of significance and n₁ + n₂ - 2 degrees of freedom. If this interval does not cover zero then the null hypothesis is rejected, otherwise it is accepted.

5 Results

5.1 Soil Morphology

5.1.1 Freeze-Thaw Cycling

The simulated weathering experiment was performed to test the hypothesis that freeze-thaw cycling combined with hygroscopic soil amendments will reduce the excessive bulk density of demolition site soils caused by high artifact content and excessive compaction. The hypothesis suggests that hygroscopic amendments can rapidly accelerate the mechanical disintegration of artifacts caused by natural freeze-thaw activity via ice-wedging and salt crystallization.

Visual examination of the soil bins after the freeze-thaw cycling experiment suggested that some of the soils had developed significant aggregation. Recent field work (Howard and Olszekska, 2011; Dubay, 2012) has shown that ^Au horizons will develop in demolition site soils in as little as 12 to 24 years.

The soil bins began to develop a weak, fine granular soil structure during the course of the experiment (Fig. 19). This result is consistent with that observed in a simulated weathering experiment by Dubay (2012). Large cracks and ridges developed on the soil surface in most of the plastic bins after 24 freeze-thaw cycles (Fig. 20). The formation of stable soil aggregates was observed through the course of the experiment. Soil aggregates generally form as a result of volume changes in the clayey materials of the soil (Brady and Weil, 2008). When soil dries out, clay mineral components move closer together causing the soil mass to shrink in volume (Brady and Weil, 2008). Cracks in the soil, as seen on the surface of the sample bins, form as the soil mass shrinks. The cracks and networks become better defined with successive cycles of wetting and drying. Note that freezing is considered a type of drying since "the formation of ice

crystals...draws water off of clay domains" (Brady and Weil, 2008). The associated swelling and shrinking of the soil with each freeze-thaw cycle creates large "cracks and fissures and pressure that alternately break apart large soil masses and compress soil particles into defined structural peds" (Brady and Weil, 2008). Figure No. 19 is a close up view of the soil surface of Bin No. 4. A well-defined system of fissures and the weak granular structure can be easily seen on the soil surface after 24 freeze-thaw cycles.



Figure 19: Close up of large fissures and weak granular structure developed in Bin No. 4.





Figure 20: Soil surface after 24 freeze-thaw cycles: A, Bin No. 1; B, Bin No. 2;, C, Bin No.3; D, Bin No. 4; E, Bin No. 5; F, Bin No. 6; G, Bin No. 7; H, Bin No. 8; I, Bin No. 9; J, Bin No. 10.

5.1.2 Wet-Dry Cycling

As with the freeze-thaw cycling, the wet-dry cycling experiment also caused significant physical changes to the simulated urban soils (Fig. 21). The repeated wetting and drying of the soil caused changes in the volume and a repeated expansion-contraction of the soil. A large system of fissures developed in the salt bins, similar to what was observed with the freeze-thaw experiment. The salt bins became massive and unhealthy in appearance. The bins treated with the sugar solution did not develop significant cracks or fissures at the soil surface during the course of the wet-dry experiment. The sugar bins developed structure and a healthy dark color. In contrast, to the sugar-treated bins, the salt-treated bins had salt crusts on the soil surface and aluminum baking pans used for the experiment experienced significant corrosion from the salt solution. The aluminum was corroded completely through the pan in some instances, as seen in Figure 22.

In general, the two soil bins treated with the sugar solution appeared much healthier than the two bins treated with the salt solution at the end of the experiment. This visual observation may be due to a healthier soil microorganism community in the sugar-treated bin. Sugar is an excellent food for microbes. It has been shown (Cernohlavkova et al., 2008) that deicing salts can limit microbial growth within soil. Previous work has also shown that increased salt levels in soil causes decreased permeability and mobilization of heavy metal ions (Amrhein et al., 1992; Backstrom et al., 2004). The bin treated with the salt solution also began to develop a pronounced "crust" of salt precipitate on the soil surface. Sugar precipitate was not observed at any point during the wet-dry cycling experiment.

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Figure 21: A, Interconnected fissures in the soil surface developed in the bin treated with a salt solution; B, Weak granular structure developed in the bin treated with a sugar solution.



Figure 22: Corrosion of the aluminum pan treated with salt solution.

5.2 Effects on Artifacts

5.2.1 Freeze-Thaw Cycling

Repeated freeze-thaw cycling of the simulated soil samples had significant effects on the buried artifacts (brick samples). Initial inspection of the brick samples after the freeze-thaw cycling revealed little physical change in most of the brick samples. Only the sand-lime samples consistently displayed severe physical breakdown and weathering (Fig. 23 and 24). In all of the soil bins, except for the bin treated with calcium chloride, the sand-lime samples were so severely degraded that post-compression testing could not be performed. This result was consistent with the control compressive strength testing, as the sand-lime bricks possessed significantly lower compressive strength values when compared with the clay and concrete bricks.



Figure 23: Severe physical breakdown of a sand-lime brick sample.



Figure 24: Spalled pieces of sand-lime brick after freeze-thaw cycling.

5.2.2 Wet-Dry Cycling

The wet-dry cycling experiments seemed to have little effect on the deterioration of the brick samples. Although one of the brick samples displayed significant spalling (Fig. 25), no other obvious signs of deterioration (cracks, spalls, pitting, etc.) were noted during visual observation. The spalled sample may have already been weakened from the initial freeze-thaw cycling and eventually split due to salt crystallization during the wetdry cycling experiments.



Figure 25: Spalled brick sample removed from salt wet-dry experiment

5.3 Compression Testing

5.3.1 Control Tests

Four control bricks of each type were subjected to compressive testing in order to establish a set of control values for comparison after the completion of the simulated weathering experiments. The table below (Table 3) summarizes the results of the compressive testing for the control samples. Mean compressive strength values for the clay bricks ranged from approximately 20 MPa to 40 MPa, with an overall mean value of 33 MPa. The concrete samples had a similar compressive strength with an average value

of approximately 33 MPa. The sand-lime brick samples possessed much lower compressive strengths, with a mean value of approximately 9 MPa. For the clay samples, the standard deviations ranged from 4.04 MPa to 16.79 MPa, while the coefficient of variation ranged from 0.13 to 0.46.

Sample	Compressive	Standard	Coefficient
ID	Strength Mean	Deviation	of
	(MPa)		Variation
В	31.41	4.04	0.13
D	29.25	10.13	0.35
IB	40.48	7.55	0.19
IIB	33.57	7.83	0.23
U	20.09	9.33	0.46
W	46.95	16.79	0.36
С	32.48	1.06	0.03
J	9.30	1.04	0.11

Table 3: Compressive strength data for control samples.

The scatterplot below (Fig. 26) shows the scatter of the compressive strengths for the control values. In general, the scatter for the control samples was fairly tight for each material type. Sample types were grouped together for the scatterplot. Although a few different types of clay brick samples were used for the experiment, they were combined to determine the scatter of compressive strength values. Figure 27 shows the frequency distribution for all control data.



Figure 26: Scatterplot showing compressive strengths for control samples.



Figure 27: Frequency distribution of all control sample data.

T-tests were performed on the control data set in order to determine the statistical significance between the brick types. The following table (Table 4) is a summary of the t-test values for the control samples.

t-values								
BRICK	В	D	IB	IIB	U	W	С	J
В	-	0.34	1.86	0.43	3.92*	2.01	0.44	14.45*
D	-	-	1.78	0.68	1.33	1.81	0.54	3.92*
IB	-	-	-	1.27	3.40*	0.7	1.78	8.18*
IIB	-	-	-	-	2.21	1.44	0.23	6.14*
U	-	-	-	-	-	2.80*	2.24	2.30
W	-	-	-	-	-	-	1.45	4.48*
С	-	-	-	-	-	-	-	28.95*
J	-	-	-	-	-	-	-	-

 Table 4: t-test values for all control sample data. * denotes a statistical significance with a confidence of at least 95%.

Based on this statistical analysis, the bricks were separated into three groups for post weathering analysis. Bricks were grouped into clay, concrete, or sand-lime categories. The clay bricks were grouped together in order to increase the n-value for this group and because the majority of the clay samples did not have compressive strength values that were statistically significant when compared with one another.

5.3.2 Post Weathering Tests

The table below (Table 5) provides a summary of the compressive strength testing results after the simulated weathering experiments were completed. The majority of the

brick samples tested displayed a significant decrease in compressive strength after 24 freeze-thaw cycles. The combined frequency distribution for all the post-weathering compressive strength data is plotted below and follows a roughly normal distribution (Fig. 28).

Brick	Mean (MPa)	Standard	Coefficient	Delta	Delta
		Deviation	Of	(MPa)	(%)
		(MPa)	Variation		
В	24.84	7.01	0.28	-6.58	-21%
D	23.31	6.31	0.27	-5.94	-20%
IB	31.17	14.20	0.46	-9.31	-23%
IIB	26.63	11.70	0.44	-6.93	-21%
U	16.72	12.76	0.76	-3.37	-17%
W	35.45	10.63	0.30	-11.50	-24%
С	28.58	14.40	0.50	-3.90	-12%
J	2.63	4.73	1.80	-6.67	-72%

Table 5: Summary of results after samples were subjected to simulated weathering experiments.



Figure 28: Frequency distribution of all post-weathering compression testing data.

Certain hygroscopic amendments appeared to increase the degradation of the brick samples more than others, as shown in the table below (Fig. 29; Table 6). It should be noted that during the course of the experiment some of the brick samples broke apart, making compressive strength testing after the weathering tests impossible. For the purposes of statistical analyses, these samples were assumed to have lost 100% of their compressive strength, and were thus assigned a strength value of 0 MPa. The two amendments which produced the most profound effect were the salt mix solution (sodium chloride, calcium chloride, and magnesium chloride) and the sugar solution (sucrose). The amendment which produced the smallest effect was the calcium chloride solution. CaCl₂ is well known as an alternative deicing agent that causes much less corrosion of concrete than NaCl. It had little effect on the artifacts, even the sand-lime brick. This probably occurred because CaCl₂ hydrates to form a portlandite-like phase (a major constituent of concrete and mortar) as follows: CaCl₂ + H₂O \rightarrow Ca(OH)₂ + HCl.

Bin	Mean	Amendment	Chemical
	(Strength Change)		Composition
1	-17.4%	Control	H ₂ O
2	-14.7%	Sodium Chloride	NaCl
3	-1.1%	Calcium Chloride	CaCl ₂
4	-24.4%	Magnesium Sulfate	MgSO ₄
5	-27.5%	Ammonium Sulfate	(NH4)2SO4
6	-27.3%	Aluminum Sulfate	Al ₂ (SO ₄) ₃
7	-32.5%	Urea	CH ₄ N ₂ O
8	-39.8%	Salt Mix	N/A
9	-42.4%	Sugar	C ₁₂ H ₂₂ O ₁₁
10	-24.2%	Humates	Fulvic Acid

Table 6: Amendment chemical compositions and their effects on artifacts.



Figure 29: Brick sample broken in situ during freeze-thaw cycling.

The chart below (Fig. 30) shows the effectiveness of each amendment on the compressive strength. The graph was normalized and shows the average percentage change in compressive strength for each amendment. Comparisons were also made for each brick type with the compressive strength changes normalized (Fig. 31).



Figure 30: Compressive strength loss by amendment.



Figure 31: Compressive strength for all tests, by sample type.

The following histogram (Fig. 32) shows the compressive strength loss of the samples arranged into categories based on the loss of strength as a percent. Samples which lost less than 10% are the in the first category, 10-20% are in the second category, 20-30% are in the third category, etc. The first category (strength loss of <10%) should be neglected for the statistical analyses. This is because this group includes a large number of samples for which strength loss is not statistically significant (i.e. losses of 1 or 2% should not be considered significant). The samples which saw of a loss of less than 10% are likely within the range of variation, based on the control testing. Instead, this study considers a significant loss of compressive strength to be a loss of 20% or greater.

By this criteria, 56% (53 out of 95) of the samples had a post-weathering compressive strength loss of at least 20%, which is considered significant by this study. The plots seen below show the comparison of compressive strength values for the control samples versus the experimental samples. The graphs (Fig. 33 through 36) are broken up by amendment (bin) and brick composition. For comparison, the clay brick samples have been combined together.



Figure 32: Compressive strength loss histogram.



Figure 33: Strength change in clay brick samples after freeze-thaw cycling.



Figure 34: Strength change in sand-lime samples after freeze-thaw cycling.



Figure 35: Strength change in concrete samples after freeze-thaw cycling.







Figure 36: Plots of control strength values versus experimental strength values for all samples and all amendments.

A *t*-test analysis was also performed for each sample type in order to verify the results of the compression testing as statistically significant. In order to increase the n value for sample types, the results of the compression testing for the individual bins were combined to generate a dataset based on sample type (i.e. concrete, clay, and sand-lime). The following table (Table 7) summarizes the *t*-test results for the experimental data set. Student's *t*-test was not able to be performed on the concrete and sand-lime samples due to n values of 1 caused by the limitations of the experimental set up. Although only 4 of the 10 results were deemed statistically significant at the 90% confidence interval, the conclusions drawn in this study are also supported by the thin section analysis and the fact that multiple brick samples spalled and fractured during the course of the experiment.

<i>t</i> -test	Bin	Bin	Bin	Bin	Bin	Bin	Bin	Bin	Bin	Bin
	#1	#2	#3	#4	#5	#6	#7	#8	#9	#10
Clay	0.52	0.86	0.82	1.49	1.50	1.82*	1.94*	3.13*	1.93*	1.23
Concrete	-	-	-	-	-	-	-	-	-	-
Sand-Lime	-	-	-	-	-	-	-	-	-	-

Table 7: t-test results of control data versus experimental data, * denotes significance at 90% confidence interval.

5.4 Electrical Conductivity and pH

Soil samples from each bin were subjected to electrical conductivity and pH testing after the simulated weathering experiments to determine if excess salinity was created by the amendments. The results (Table 8) show that the amendments caused a negligible change in pH. There was either no change, or a slight decrease in pH for some amendments. Salinity effects on plants are usually negligible at an EC < 2,000 μ S cm-1 (Bower and Wilcox, 1965). Hence, the only amendment which would potentially have an adverse effect on plant growth was the salt mixture. These relationships are attributable to the relatively low (3%) concentrations of amendments used in the experiment.

Bin	Amendment	рН	Electrical Conductivity
			(µS/cm)
Control	N/A	7.71	867
1	Water	7.39	1,180
2	Sodium Chloride	7.57	1,810
3	Calcium Chloride	6.97	1,100
4	Magnesium Sulfate	7.55	656
5	Ammonium Sulfate	7.16	1,511
6	Aluminum Sulfate	7.39	1,398
7	Urea	7.57	1,051
8	Salt Mix	7.33	3,780
9	Sugar	7.57	742
10	Humic Acid	7.69	762

Table 8: Electrical conductivity and pH measurements.

5.5 Thin Section Analysis

The results of point counts of thin sections of brick samples that underwent both the freeze-thaw cycling and wet-dry cycling simulated experiments are shown in Table 9.

Sample	Туре	Treatment	Mineral	Pore	Mineral	Pore %
					%	
B-1	Concrete	Control	366	34	92%	9%
B-2	Concrete	Sugar	349	51	87%	13%
B-3	Concrete	NaCl	357	43	89%	11%
B-4	Clay	Control	381	19	95%	5%
B-5	Clay	Sugar	358	42	90%	11%
B-6	Clay	Control	384	16	96%	4%
B-7	Clay	NaCl	371	29	93%	7%

Table 9: Thin section point-counting results.

The results suggest that microporosity was increased in each sample type after treatment with either sugar or NaCI. This is consistent with the results of the compression testing. The freeze-thaw cycling combined with the various treatments seems to have caused increased stresses in the brick samples, leading to microcracks and increased pore space. The cycle appears to be a positive feedback system so that as more cracks and pore space were created, there was more room for water to penetrate the brick and freeze, causing more internal damage to the sample.

The photomicrographs show an increase in microcracks and pore space after the weathering experiments (Figure 37). The samples were injected with blue dye, hence the pore space appears blue in the following photomicrographs.

Although the strength tests showed a consistent decrease in strength and point counting showed a consistent increase in microporosity, the results in Table 10 show that only the clay brick amended with sugar can be shown to have a statistically significant increase in microporosity. This is interpreted to mean that although the differences are thought to be statistically significant, the small sample size precludes an accurate statistical test. Unfortunately, the number of samples which could be analyzed was limited by this and is thought to be the result of limited numbers of samples. Due to logistics of carrying out the study, only a limited bin size could be studied, which therefore limited the number of bricks used per bin.

To evaluate the point counting, *t*-tests were also performed on the point counting results in order to determine the validity of the data. The results of the *t*-test analysis show that three of the four comparisons were not statistically significant with a confidence interval of 95% (Table 10).

 Table 10: Tests for statistically significant differences in microporosity between samples before and after freeze-thaw/wet-dry treatments.

Sample pair	95% confidence intervals (p1 - p2)	Hypothesis (p ₁ = p ₂)
	Concrete Brick	
Control vs. Sug Control vs. Nac	ar $-0.003 < p_1 - p_2 < 0.083$ Cl $-0.022 < p_1 - p_2 < 0.062$	Accept Accept
	Clay Brick	
Control vs. Sug Control vs. Na	par 0.023 <p1-p2 0.097<br="" <="">-0.002 <p1-p2 0.062<="" <="" td=""><td>Reject Accept</td></p1-p2></p1-p2>	Reject Accept




Figure 37: Photomicrographs of brick sample thin sections as a function of treatment. A, concrete control; B, concrete with sugar treatment; C, concrete after NaCl treatment; D, Brick type 1 control; E, Brick type 1 after sugar treatment; F, Brick type 2 control; G, Brick type 2 after NaCl treatment.

6 Discussion

The results of this study are consistent with field observations that brick is much more durable than mortar in masonry structures (Hughes and Bargh, 1980). Howard et al. (2013) found that disintegration of mortar artifacts was widespread in urban Detroit soils 30 to 100 years old. They showed petrographic evidence for increased microporosity, although they attributed this to chemical dissolution based on the development of ^Ck horizons. The petrographic results presented here suggest that at least some of this microporosity may be the result of mechanical disintegration caused by freeze-thaw cycling. The sand-lime bricks (simulating mortar) were clearly weakened considerably during the freeze-thaw experiment. Indeed, building codes in Detroit now prohibit the use of this type of sand-lime brick due to its propensity to weaken and fail because of surface weathering. In this study, the process was enhanced further through the use of hygroscopic amendments. In the case of concrete, the photomicrographs suggest that the disintegration process occurred as calcite grains were dislodged from limestone aggregate in the concrete (Fig. 37) by freeze-thaw activity.

Modern bricks are typically fired at > 900° C, thus forming a surface coating of glass (Cultrone et al., 2004). Hence, such bricks are highly resistant to decomposition by chemical weathering. Instead, brick is well known to disintegrate mechanically by the spalling-off of platy fragments. The results of this study suggest that this spalling process is initiated as silt-sized quartz grains are dislodged by freeze-thaw thereby creating a system of voids (Fig. 37). These voids presumably enlarge until spalling occurs on a macroscopic scale.

It is well known that excess soil salinity can have an adverse effect on plant growth (Cunningham et. al., 2007; Ramakrishna and Viraraghavan, 2005). The results obtained

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suggest that excess salinity would not be an issue if hygroscopic amendments were used in the field. It may also be possible to use higher concentrations of amendments in the field because no leaching took place in this experiment. Dubay (2012) showed that extensive leaching of salts took place during his simulated weathering experiment. He found complete dissolution of drywall artifacts after simulating 30 years of chemical weathering. The use of dilute solutions containing hygroscopic compounds is also unlikely to adversely affect groundwater quality.

The type and abundance of artifacts generally found in urban soils depends on the age and type of building construction, the demolition methods used by a particular contractor, and changes in demolition regulations over time. Generally, the most common artifacts in urban soils of Detroit are brick, mortar and concrete. In the older part of Detroit, demolition site soils contain bricks from 19th century buildings (Howard et al., 2013). These bricks were generally fired at temperatures < 900°C, hence they lack a surface coating of glass and are more porous. Thus, the use of hygroscopic amendments to enhance the effects of freeze-thaw may be even more effective on soils containing these older types of bricks.

7 Conclusions

The results of this study support the hypothesis that dilute solutions containing hygroscopic compounds can be added as amendments to demolition site soils to affect the mechanical breakdown of rock-like artifacts in situ. The method also appears to be feasible on a time scale (< 10 years) short enough to be useful in the near future, and thus provides a more cost-effective alternative to physical removal. Although many of the compounds studied were salts, they did not result in salinity levels high enough to adversely affect groundwater or plant growth. It is likely that with further study a cocktail of hygroscopic compounds could be concocted which would simultaneously provide other service functions. For example, sugar could be used to promote microbial activity, fertilizer salts to enhance fertility, and organic compounds to promote aggregation and water-holding capacity. Further study is also needed to examine the possibility of recycling some hygroscopic waste material (e.g., cellulose waste generated by the paper manufacturing industry). In addition, it has been shown that a reduction in soil compaction (bulk density) may accelerate the release of soil nutrients and organic matter (Henry, 2006; Schmidt and Lipson, 2004) suggesting that soil treatments such as this may aid in the formation of a reclaimed soil suitable for urban agriculture.

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ABSTRACT

EVALUATION OF HYGROSCOPIC SOIL AMENDMENTS AND NATURAL FREEZE-THAW CYCLING TO ACCELERATE THE MECHANICAL BREAKDOWN OF ARTIFACTS IN DEMOLITION SITE SOILS

by

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Many cities worldwide have areas of vacant land produced by building demolition. This open space has attracted great interest as a potential resource for green infrastructure, urban agriculture, and other purposes related to urban renewal. Unfortunately, rock-like artifacts (e.g. brick, mortar, concrete) are often present in great abundance in demolition site soils. These artifacts make the soil difficult to till, create obstacles for root penetration, and limit the soil's water-holding capacity, infiltration and aeration. As an alternative to physical removal, this study was carried out to test the feasibility of using hygroscopic compounds as soil amendments to accelerate the mechanical disintegration of these artifacts in situ using natural freeze-thaw/wet-dry processes. Bench-scale tests were carried out to simulate 5 to 8 years of freeze-thaw/wet-dry cycling using dilute (3%) solutions of deicing salts (NaCI, CaCI2, salt mixture), fertilizer salts (MgSO4, Al2(SO4)2, (NH4)2SO4), and organic compounds (CH4N2O, C12H22O11, humate), which were added to a commercial topsoil containing clay bricks, concrete and sand-lime brick (simulating mortar). In the absence of visible

disintegration, the effectiveness of each treatment was evaluated by uniaxial compression testing, and petrographic analysis of microporosity. Sand-lime brick showed visible disintegration by all treatments except CaCl2. The clay and concrete bricks generally lacked visible signs of deterioration, but showed a consistent decrease in compressive strength with all treatments. The greatest loss in strength (30%) occurred with the use of urea, sugar and the salt-mixture; however humate and all three sulfate compounds produced an average loss in strength of 20% or more. Petrographic analysis of concrete and clay bricks treated with NaCl and sugar showed an increase in microporosity which is inferred to be the cause of strength loss. Microporosity in the concrete was due to dislodging of calcite grains in the limestone aggregate, whereas that in clay bricks resulted from dislodging of silt-sized quartz temper. The treatments had no significant effect on pH, and electrical conductivity measurements showed that the salinities of all soils (except treated with the salt-mixture) were below a level detrimental to plant growth. These results support the hypothesis tested that hygroscopic compounds can be added as amendments to demolition site soils to accelerate and effect the mechanical breakdown of artifacts on a time scale (< 10 years) short enough to be useful in the near future.

AUTOBIOGRAPHICAL STATEMENT

After completing high school, Phillip Backers began his undergraduate studies at Michigan State University in the fall of 2007. His interests in earth science and the environment led him to the Geology Department at MSU. Mr. Backers served as the Vice-President of the MSU Geology Club during his senior year, coordinating a field course in Ireland during this time. He graduated from MSU in 2011 with a Bachelor of Science Degree in geological sciences. He was then hired full-time by a regionally known civil and environmental engineering firm, NTH Consultants, Ltd., as a staff geologist working in the geotechnical engineering group. Here Mr. Backers honed his skills in the field, becoming experienced in soil borings, rock cores, and aquifer analyses. After working full-time for about a year, Mr. Backers decided to return to the classroom and pursue a Master of Science Degree part-time at Wayne State University. He began taking classes part-time and working with Dr. Jeffrey Howard to study urban soils in Detroit. Mr. Backers is looking forward to putting his newly acquired knowledge to use as he embarks on the next chapter of his professional career.