Effect of a hydrophobic layer on the upward movement of water under freezing conditions

by

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Abstract

Frost heave is the process in which wet soil with an available water source undergoes freezing, deformation, and upward movement of the soil surface. This deformation can cause damage to engineering structures such as pavements and shallow foundations. Investigating ways to minimize frost heave by reducing water flow in the system is beneficial. A possible way to reduce the water movement is to add a hydrophobic layer of soil between the water source (e.g. water table) and the freezing surface. The objective of this study was to examine the effect of a hydrophobic treated soil layer on water movement and temperature changes in a soil profile under surface freezing conditions. A vertical soil cell set-up including a column-within-a-column design was used to establish one-dimensional vertical heat flow between a surface boundary condition below freezing and an ambient boundary temperature condition at the bottom of the cell. A constant water table was established at the bottom of the column to provide a water source for water uptake due to freezing. Water uptake in soil cells without a hydrophobic layer was found to be greater by one order of magnitude than water uptake in soil cells with a hydrophobic layer. Soil with a hydrophobic layer had less accumulation of ice and froze to greater depths than soil without a hydrophobic layer. A hydrophobic soil layer can reduce water movement in freezing soil.
Introduction

Of particular interest in the fields of engineering, agronomy, and earth science is the movement of water due to freezing of soil and the resulting upward movement of the soil surface, commonly referred to as frost heave. Frost heave can cause damage to a variety of man made structures (e.g. shallow building foundations) and is extremely detrimental to the performance and safety of roadways, both paved and unpaved. In an agricultural setting, frost heave can cause damage to the roots of perennial plants resulting in losses to production. Thus, there is value in studying frost heave and developing methods to reduce its effects.

Frost heave in soil has long been observed, but it is only in the past 80 years that studies have documented the mechanisms through which it occurs. Taber (1929, 1930) described the increase in volume due to freezing of soil and recognized the role of liquid movement toward a freezing front. Taber showed through laboratory experiments that soil wetted with liquids that contract upon freezing still heave under continuous freezing conditions. This study was important as the volume of frost heave often seen in the field goes beyond what can be accounted for from the 9% volume expansion of water during the phase change from liquid to solid. While water expansion does affect the system, the primary mechanism of frost heave is water movement to a freezing front and accumulation as ice. Beskow (1935) confirmed the finding by Taber (1929, 1930) of water movement in the soil capillary system. Liquid water exists in thin films between the ice and soil particles allowing liquid water to move in this interface.

The flow of water toward a freezing front is often described as similar to the effect of drying soil. Some studies suggest that the water movement toward a freezing front results
from surface tension at the contact between ice and unfrozen water in pores (Evert 1961) and
the development of pore water pressure gradients related to this contact (O’Neill 1985).
Some suggest that the repulsion of ice and soil in pores causes the development of films
between soil and ice particles (Dash 1989). Further investigation of repulsion phenomena
predicts that water movement is also associated with this repulsion between ice and soil,
resulting in decreased pressure between soil and ice and subsequent water movement toward
the low pressure (Rempel 2004). Because water movement is an important mechanism for
frost heave, treatment of soil to reduce water movement may reduce frost heave.

Sage (1993) conducted work with soil material that had been hydrophobized to
investigate whether frost heave could still occur. Sage (1993) used soil columns frozen from
the bottom and determined that ice crystals and ice lenses could form in a system of
hydrophobized fine grained soil, which had available water in the system. Sage (1993)
describes a water layer existing between the ice and soil particles with continued freezing of
water on the ice surface resulting in ice lens growth. However, the study of Sage (1993)
differed from natural field conditions because the columns were frozen from the bottom. In
addition, the water supply for ice lens formation came from the entire volume of
hydrophobized soil. In natural conditions soil freezes from the surface downward, and the
soil is usually wettable with possible hydrophobic layers. Also, water is typically supplied
for frost heave from both a water table and the unsaturated bulk soil. Reduction of water
movement from a water table toward the freezing front by altering soil wettability (i.e.,
making the soil hydrophobic) may offer a means to reduce frost heave and warrants further
investigation.
Methods to hydrophobize soil have been investigated for their effect on soil properties, especially wettability. Bachmann et al. (2001) treated soil with Dichlorodimethylsilane (CMS) resulting in hydrophobic soil. By varying the CMS-application amount various contact angles were investigated to determine the amount of CMS needed to produce an extremely water repellent (i.e. large contact angle) soil. The CMS-method provides a relatively simple means to reduce soil wettability and potentially limit water flow.

Natural field conditions for frost heave involve heat and water transfer that is predominantly one-dimensional (1-D). Water moves from a water table toward a freezing front extending from the soil surface. Laboratory column studies to examine coupled heat and water transfer in soil have often been reported, but 1-D temperature gradients are often difficult to achieve. Ambient temperatures in the laboratory can produce two-dimensional temperature gradients (Prunty and Horton, 1994). Zhou et al. (2006) developed a soil cell consisting of a small inner column of unfrozen soil surrounded by a larger column of the same soil to provide insulation in order to reduce ambient temperature interference and achieve 1-D conditions. The advantage of this cell is the replication of materials and conditions in both the inner and the outer columns. This reduces the effect of changes to soil thermal properties that occur related to water movement in soil and provides an optimized volume of soil in the inner column for measurements to be taken. This cell used spiral heat exchangers located at both ends of the soil cell. While this cell design provides opportunity to more closely match 1-D field conditions, it has not been tested for the investigation of soil under a freezing condition. Also, the use of chemical treatments to reduce water movement
associated with freezing has not been intensively investigated. Increasing this understanding could lead to new methods to reduce frost heave.

The objective of this study was to investigate the influence of a hydrophobic soil layer on water redistribution in soil undergoing surface freezing conditions with 1-D heat flow and an available basal water source. Also investigated were the soil temperature changes at various depths related to the water movement. It was expected that the large contact angle of the hydrophobic soil layer would reduce upward water movement through the capillary system as compared to wettable soil.

Hypotheses of this study were 1) Less water would move into the soil cells that contained a hydrophobized layer as compared to cells without a hydrophobic layer. 2) Because the soil had an initial amount of liquid phase water in the matrix (i.e., available water) water would redistribute in all soil cells toward the freezing front resulting in a mass accumulation of ice. 3) The amount of ice formed would be reduced in the cells with a hydrophobic layer compared to the soil cells without a hydrophobic layer due to reduced water uptake. 4) Increased depth of freezing in soil cells containing a hydrophobic layer compared to soil cells without a hydrophobic layer due to reduced water uptake, reduced soil heat capacity and smaller release of latent heat from ice formation.
Materials and Methods

Soil Material and Hydrophobizing Treatment

A naturally hydrophilic soil, Ida silt loam (Fine-silty, mixed, superactive, calcareous, mesic, Typic Udorthents) was used in the experiments. Soils containing a large amount of silt sized particles subject to a small surface confining force can exhibit a large amount of frost heave in comparison to soils that contain a greater amount of coarse (sand and gravel) or fine (clay) sized particles (Miller et al. 1980). Ida silt loam was used because of its high silt content. The soil was collected from the loess hills region of Western Iowa, air-dried, ground, and sieved to a size of \( \leq 2 \) mm.

A portion of the soil was rendered hydrophobic following the technique used by Bachmann et al (2001). Dichlorodimethylsilane (CMS) was used as the hydrophobizing agent. The technique for hydrophobic treatment of the soil was as follows;

1. Soil was divided into small batches of 100 g and placed in a fume hood.
2. 4.8 ml of CMS was added to each batch of soil using a pipette.
3. The mixture was stirred until all the CMS had been dispersed through the material and any clods that had developed upon addition of the CMS were broken.
4. Each sample was allowed to set for at least one hour with periodic stirring to ensure even distribution of CMS.
5. Deionized water was added to fully immerse the sample.
6. The sample was air dried in the fume hood.
7. Air dried batches were combined to provide a uniform hydrophobized soil.

The contact angles of the untreated and the CMS-treated soils were determined by the Wilhelmy plate method (Bachmann et al., 2003). Wettability of the untreated and CMS-
treated soil was measured using the water drop penetration time test (WDPTT). The WDPTT determines the time required for a surface applied water drop to infiltrate into soil (Dekker and Ritsema, 1994).

**Soil Cells**

A soil cells was designed to produce 1-D heat flow toward the freezing conditions at the upper boundary. The design of the soil cell was similar to that used by Zhou et al (2006). The cell consists of a column-within-a-column design. In this study, both the inner and outer soil columns were constructed from PVC with 20-cm length. The internal columns had 3.8-cm diameter, while the outer soil columns had 8.9-cm diameter. Figure 1 shows a detailed schematic of the soil cell design. Insulation was provided by filling the larger surrounding column with the same soil type at the same initial density and moisture conditions used in the internal column. In addition, a layer of Reflectix® bubble insulation (Reflectix Inc., Markleville, IN) surrounded by 3.8 cm thick fiberglass pipe jacket insulation (Insulation World, Hopewell, VA) was wrapped around the soil cells.

Soil cells were oriented with the long axis vertical during experiments. The upper boundary temperature for the soil cells was established with a subfreezing surface condition using a liquid cooled heat exchanger as shown in Figure 2. A 1:1 mixture of ethylene glycol and water was cooled using a temperature controlled bath (Programmable Digital Circulator, Model 9512, PolyScience, Niles, IL) and pumped though the heat exchanger. The cooled fluid entered the heat exchanger at the center and circulated in a spiral flow pattern across a thin (0.5 mm) copper plate that was in contact with the entire soil cell surface and exited the heat exchanger on the outer edge. The design of the heat exchanger ensured that the entire upper surface of the soil cell was exposed to near uniform temperatures. After exiting the
heat exchanger, the fluid circulated to the temperature controlled bath and cooled in the reservoir to the set temperature. A separate heat exchanger was placed on the top of each soil cell and sealed and secured with o-rings which would maintain contact of the heat exchanger with the soil surface yet not induce a large confining force allowing upward movement of the soil surface. The lower boundary temperature was the ambient temperature condition. Figure 3 shows a photo of the system in the laboratory with all connections in place at the beginning of an experiment.

Connections were made to establish a water table on the bottom of all soil cells; water was maintained at ambient conditions and was allowed to flow into or out of the cells to maintain this water level. The volume of water entering each internal column was recorded using Marriott style calibrated burettes. The external insulating column water source came from a separate Marriott bottle.

Each soil cell was instrumented with 7 copper-constantan (Type T) thermocouples which were placed in the inner column to record the soil temperature over time for observation of the freezing front depth. Thermocouples were placed at the surface and depths of 2 cm in center of column, 4 cm in center of column, 6 cm in center and edge of column, 10 cm in center of column, and 14 cm in center of column. Placement of thermocouples at both the center and edge allowed comparison of radial versus longitudinal temperature gradients to assess whether 1-D conditions were achieved. Temperature data were collected each minute and averaged each 30 minutes for data analysis using a datalogger (model 21X, Campbell Scientific, Logan, UT) and a multiplexer (model AM16/32, Campbell Sci.).

**Experimental Conditions**
Two experiments were performed with four soil cells used in each experiment. Two soil cells contained hydrophobic treated soil layers and two soil cells did not. Heat exchangers were connected to the cooling baths by Tygon® flexible wall tubing from the cooling bath. The length of the tubing connecting each heat exchanger to the water bath was considered and effort was made to make the flow distance equal for each set of heat exchangers to eliminate differentials in surface conditions due to heat transfer though flexible wall tubing. Pieces of flexible wall foam tubing were fitted over the Tygon® flexible wall tubing to reduce the amount of heat transfer to the cooling liquid in the circulation path to heat exchangers.

Moist soil was packed into the cells in 10 lifts of 2 cm each to a dry density of 1.25 g cm\(^{-3}\). The soil was wetted prior to packing with 0.05 mmol CaCl\(_2\) to achieve a mass water content of 25 % resulting in a 60 % degree of saturation. Soil was packed to the proper density using a cylindrical tamping rod with a surface area of 0.75 cm\(^2\). The layer of hydrophobic treated soil was placed in two of the soil cells at a depth of 16 to 18 cm and a dry density of 1.25 g cm\(^{-3}\); this hydrophobized soil had an air dry moisture content of approximately 4% resulting in a 10 % degree of saturation. In the remaining two cells, a corresponding layer of air-dry untreated soil was placed at the 16 to 18 cm depth. The external column was filled with soil of the same type and in the same condition as that which was inside the inner column. Matching layers of hydrophobic treated soil and untreated soil were located at the same depths in the external column as those in the internal column.

A water table was imposed on each soil cell in the bottom 1 cm section (depth of 19 to 20 cm), and water uptake was allowed to occur for 24 hours before reducing the surface temperature. After 24 hours, the upper boundary of the soil cell was decreased to -5° C using
the temperature controlled bath. This temperature was maintained by the temperature controlled bath throughout the experiment. The lower boundary condition was subject to ambient temperature conditions with the water supply for the water table. Water uptake into the inner column from the water table was recorded for the duration of the experiments. Two experiments were performed. Experiment 1 had an ambient temperature of 24° C and was maintained for 550 hours. Experiment 2 was performed in a climate controlled growth chamber with an ambient temperature of 10° C, to impose lower temperature gradients than in Experiment 1, and was maintained for 1100 hours.

Upon completion of experiments, calibrated calipers were used to determine frost heave for each cell was by measuring expansion of the soil above the original internal soil column boundaries. Visual description and photographs were taken of the external soil column as the soil cells were dissected. Soil from the inner soil columns was removed in 2 cm intervals. This material was weighed, dried at 105 °C for 24 h, and re-weighed to determine the final water content distribution within each internal soil column. Statistical analysis of data for comparison of treatments was performed using a Students t test (Ott and Longnecker, 2001) with the JMP software package (Version 5.1.2, SAS Institute, Cary, NC).
Results and Discussion

Soil Treatment

The contact angles of untreated and CMS-treated soils were $0^\circ$ and $130^\circ$, respectively. Based on contact angle measurements, the untreated soil was wettable and the CMS-treated soil was hydrophobic. The WDPTT results for water drop infiltration times were $< 5$ s for the untreated soil and $> 3600$ s for the CMS-treated soil. The untreated soil was classified as wettable and the CMS-treated soil was classified as extremely water repellent (Ritsema and Dekker, 1994; Letey et al., 2000). Figure 4 shows untreated soil and CMS-treated soil 15 seconds after a water drop was placed upon each soil. The water drop wetted the untreated soil while the water drop remained in place on top of the CMS-treated soil.

Experiment 1

Upon circulation of freezing temperature fluid in the system, surface temperatures were lowered to below $0^\circ$ C in all cells in less than 1 hour. This ensured that the testing apparatus was working properly and that below freezing conditions would be maintained at the surface. The liquid cooling baths were set at a temperature of $-6^\circ$ C and all of the soil cells had surface temperatures between $-5^\circ$ C and $-6^\circ$ C. Temperature of the fluid in the bath was shown by digital display on the cooling bath and confirmed with an alcohol thermometer placed in the cooling fluid. Final vertical mean boundary temperature gradient for the cells was $1.45^\circ$ C cm$^{-1}$ and the mean radial gradient at a depth of 6 cm was $0.21^\circ$ C cm$^{-1}$. This is a vertical to radial temperature ratio of 7:1. This ratio shows that predominant heat flow was in the vertical direction, and 1-D heat transfer conditions were established in the cells. Thus,
the objective that one dimensional heat transfer conditions would be established was supported experimentally.

Final water content distribution profiles and water uptake amounts indicated that the soil cells with a hydrophobic layer had lower amounts of water uptake compared to soil cells without a hydrophobic layer. Analysis of the water uptake volumes can be seen in Table 1. The hydrophobic layer caused much less water to enter cells because restricted water flow through toward the freezing front.

Cells containing a hydrophobic layer had lower mean water content and lower maximum water content than the cells without a hydrophobic layer. Cells without a hydrophobic layer had maximum soil water contents of 102% and 170%. Cells with a hydrophobic layer had maximum soil water contents of 53% and 46%. These peak values were found in ice lenses at the freezing fronts and can be seen when the data are graphed with depth as shown in Figure 5. Visual inspection of dissected cells showed the hydrophobized soil layer starting at a depth of 16 cm was dry and ice accumulation in untreated cells was much greater. Water was redistributed in all cells toward the freezing surface. Cells with a hydrophobic layer experienced decreased mass water content at depths between 6 cm and 16 cm as water above the hydrophobic layer moved toward the freezing surface to increase water contents in the top 6 cm. While highly nonwettable and dry in appearance, some water was able to enter the hydrophobic layer and moisture content increased from an initial value of 4% to an average mass water content of 19%.

Figure 6 shows a temperature versus time data series obtained from the thermocouples in a soil cell without a hydrophobic layer during Experiment 1. Although the actual temperature values measured in the other soil cells differ slightly from the values
shown in Figure 6, the trends shown in Figure 6 are representative of the temperature versus
time data measured in all of the soil cells. All of the soil temperature values responded to the
imposed sub-freezing surface boundary condition. The measured temperature values
decreased in an orderly fashion beginning with the shallow depths and proceeding to deeper
soil. All of the temperature values decreased rapidly in the first 12 hours after initiation of
surface cooling. After 4 days the temperature values were quite stable and were asymptoting
to the final temperature distribution.

As hypothesized, temperature decreased more rapidly in soil with a hydrophobic
layer than in the soil without a hydrophobic layer. Table 2 shows the time required to
decrease temperature to 0°C at various depths in the profile. While not statistically different
because of variability in the measurements, average time to reach 0°C was greater for cells
without a hydrophobic layer. On average, soil temperatures were lower throughout the
profile of cells containing a hydrophobic layer and ice formation was deeper. Figure 7 shows
final temperature profiles for two soil cells in Experiment 1. One reason for the difference in
freezing depth is that soil containing less water, such as that found in soil cells with a
hydrophobic layer, has a lower heat capacity. Another reason is that increases in soil water
content due to water uptake, such as in soil cells without a hydrophobic layer, results in
added heat associated with the water as well as an increased release of latent heat due the
greater amounts of ice formation.

Frost heave occurred in both soil cells but was less in soil cells containing a
hydrophobic soil layer. Soil cells without a hydrophobic layer heaved 0.9 cm, and cells with
a hydrophobic layer heaved 0.3 cm.

Experiment 2
Data obtained in Experiment 2 with lower ambient temperature conditions was consistent with that from Experiment 1. Upon circulation of freezing temperature fluid in the system, surface temperatures were lowered to a subfreezing condition in all soil cells in less than 1 hour. The liquid cooling baths were set at a temperature of -6°C and surface temperature for all soil cells was below -5°C. The final mean vertical temperature gradients for the soil cells was 0.76°C cm⁻¹, which is less than in Experiment 1, and the mean radial gradient at a depth of 6 cm was 0.17°C cm⁻¹, which is similar to the value obtained in Experiment 1. This is a vertical to radial temperature of 5:1; this ratio shows heat flow was predominantly in the vertical direction and 1-D heat transfer conditions were established.

Analysis of the water uptake volumes can be seen in Table 1. The hydrophobic layer caused much less water to enter soil cells. This was similar to the effect seen in Experiment 1. The ambient temperature difference had little effect on the amount of water uptake by the cells, and the water uptake volumes were similar at equivalent times of freezing.

Soil cells containing a hydrophobic layer had lower mean water content and lower maximum water content in the profile compared to soil cells without a hydrophobic layer. Soil cells without a hydrophobic layer had maximum water contents of 100% and 122%. Soil cells with a hydrophobic soil layer had maximum water contents of 40% and 42%. These peak values were found in lenses at the freezing fronts and can be seen when the data are graphed with depth as shown in Figure 8. When visually examined, hydrophobized layers appear dry in comparison to soil layers above and below and ice lenses are smaller or not visible in cells with a hydrophobic layer, see Figure 9. Water was redistributed in all cells toward the freezing surface. Cells with a hydrophobic layer had decreased mass water content at depths between 10 cm and 16 cm as water moved toward the freezing surface to
increase water contents from the top 10 cm. Water was able to enter the hydrophobic layer and the water content in the layer was increased from the initial value of 4% to 23%. Maximum water content was seen at the same depth, 8 cm, in both treated and untreated cells.

The rate of the freezing front penetration was faster in the cells with a hydrophobic layer. The rate of penetration of the freezing front was also greater at the lower ambient temperature of Experiment 2 than for the larger ambient temperature of Experiment 1. Table 2 shows the time required to decrease temperature to 0°C at various depths in the soil cell profile. The soil froze to greater depths in Experiment 2 than in Experiment 1. Figure 10 shows temperature profiles for two soil cells at the conclusion of Experiment 2. Average soil temperatures were lower throughout the profile of cells containing a hydrophobic layer, and ice lens formation was deeper when compared to cells without a hydrophobic layer.

Very little frost heave occurred in any of the cells, and there was no difference in the amount of frost heave observed in soil cells regardless of treatment.
Summary and Conclusions

The objective of this study was to develop a system that approximated 1-D heat flow under surface freezing conditions and to observe the effect of an introduced hydrophobized soil layer on water movement from the water table and on temperature distributions. Replicated laboratory experiments were performed under two different ambient temperature conditions. The first hypothesis of this study, that less water would move into the soil cells that contained a hydrophobic soil layer, was confirmed. Water uptake was less in soil cells with a hydrophobic soil layer than in cells without a hydrophobic layer. The second hypothesis of this study, that in all soil cells water would redistribute toward the freezing front resulting in a mass accumulation of ice, was confirmed. Increases in water content were observed in the profile of all soil cells near the surface. Soil cells with a hydrophobic layer had reduced water contents from the initial water content between the ice lens and the hydrophobic layer indicating redistribution. The third hypothesis of this study, that less ice would form in soil columns containing a hydrophobic layer, was confirmed by visual observation of the dissected soil columns and by profile mass water content measurements. Soil cells without a hydrophobic layer had greater water uptake from the water table allowing for a greater accumulation of ice. The fourth hypothesis of this study, that soil cells containing a hydrophobic layer would freeze deeper than those without a hydrophobic layer, was confirmed. Compared to soil cells with a hydrophobic layer, heat capacity and release of latent heat was greater in soil cells without a hydrophobic soil layer due to the greater uptake of water and greater ice formation. This resulted in reduced depth of freezing in the cells without a hydrophobic layer.
Future Work

Developing a field scale technique for hydrophobizing soil may be a viable spot treatment for reducing water movement in problem soils related to frost heave. In an engineering application the investigation of possible in situ treatments could be useful. Continuation of this work investigating effects of hydrophobic layers in different soil types would be of interest. This type of investigation would be needed for successful field experiments of chemically hydrophobizing soil to reduce water movement in frost susceptible soils and locations.
References Cited


Ott, R.L., and Longnecker, M. 2001. *An Introduction to Statistical Methods and*


Table 1. Cumulative water uptake of soil cell inner soil columns as a function of time.

<table>
<thead>
<tr>
<th>Layer Classification†</th>
<th>Cumulative water uptake</th>
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<td>Day 3</td>
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<td>Day 17</td>
</tr>
<tr>
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<td></td>
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<tr>
<td>Wettable</td>
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<td>Growth chamber (Experiment 2)</td>
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</tbody>
</table>

†Values listed for each layer include two replicates.
*Values within grouping were statistically different by Student’s t test (alpha = 0.05).
Table 2. Time required for specific soil depths to cool to 0°C.

<table>
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<th>4 cm</th>
<th>6 cm</th>
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<td>13.9</td>
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†Values listed for each layer include two replicates.
Figure 1. Cross-section of the column-within-a-column soil cell.
Figure 2. Components of the heat exchanger used for controlling the soil cell upper boundary temperature.
Figure 3. The soil cell arrangement during Experiment 1.
Figure 4. Untreated (left) and CMS-treated (right) soil during water drop penetration time test (WDPTT), elapsed time was 15 seconds.
Figure 5. Mass water content profiles in the inner soil columns at the conclusion of Experiment 1. The ambient temperature was 24° C and the surface boundary temperature was -5° C during the experiment.
Figure 6. Temperature with time data obtained from thermocouples placed at different depths in the soil cell. The trends are typical of all of the soil cells used in Experiment 1.
Figure 7. Final temperature distributions at the conclusion of Experiment 1 for a soil cell with a hydrophobic layer and a soil cell without a hydrophobic layer.
Figure 8. Mass water content profiles in the inner soil columns at the conclusion of Experiment 2. The ambient temperature was 10° C and the surface boundary temperature was -5° C during the experiment.
Figure 9. Untreated (left) and hydrophobized (right) soil profiles in the outer soil column at the conclusion of Experiment 2. The light-colored soil layer (right) is the dry hydrophobic treated layer. An ice lens is visible in the untreated profile (left), but not in the profile containing the hydrophobized layer (right).
Figure 10. Final temperature distributions at the conclusion of Experiment 2 for a soil cell with a hydrophobic layer and a soil cell without a hydrophobic layer.