Engineered Water Repellency for Mitigating Frost Action in Iowa Soils

Ty Brooks, S.M.ASCE[1]; John L. Daniels, P.E., F.ASCE[2]; Micheal Uduebor[3]; Bora Cetin, Ph.D., M.ASCE[4]; and Mohammad Wasif Naqvi[5]

[1]MSCE Student, Dept. of Civil and Environmental Engineering, Univ. of North Carolina at Charlotte, Charlotte, NC. Email: cbrook63@uncc.edu
[2]Professor and Chair, Dept. of Civil and Environmental Engineering, Univ. of North Carolina at Charlotte, Charlotte, NC. Email: jodaniel@uncc.edu
[3]Ph.D. Student, Dept. of Civil and Environmental Engineering, Univ. of North Carolina at Charlotte, Charlotte, NC. Email: muduebor@uncc.edu
[4]Associate Professor, Dept. of Civil and Environmental Engineering, Michigan State Univ., East Lansing, MI. Email: cetinbor@egr.msu.edu
[5]Graduate Research Assistant, Dept. of Civil, Construction, and Environmental Engineering, Michigan State Univ., East Lansing, MI. Email: naqvimo1@msu.edu

ABSTRACT

Organo-silane (OS) modification has been shown experimentally to render soil water repellent. Engineered water repellency has the potential for widespread use in geotechnical and geoenvironmental applications. One such application is mitigating the effects of frost action in susceptible subgrade soils, particularly for unbound, unsurfaced roads. As part of a larger project to evaluate the feasibility of post-construction treatment of such roads, testing was performed with two commercially available OS products at varying dosages. This paper summarizes experimentally observed relationships between treatment, apparent contact angle, and water entry head for four samples of frost-susceptible soil collected from different regions across Iowa. Soil water characteristic curves were used to estimate the prevailing pore size and to relate that to water entry head for a given sample. Modification with two different OS chemicals yielded apparent contact angles between 119° and 143°. The water entry head was determined to range from 15 to 63 cm of water. Inverse relationships were observed between average pore radius and water entry head. These results are expected to be useful to agencies interested in evaluating engineered water repellency for use in new construction as well as rehabilitation of existing infrastructure underlain by frost susceptible soils.

INTRODUCTION

Engineered water repellency (EWR) has been reported to have potential for wide scale use in geotechnical and geoenvironmental applications (Kim et al. 2015; Jordan et al. 2017; Keatts et al. 2018). For example, rather than using a physical barrier (e.g., geomembranes, compacted clay) to resist water infiltration, in situ soils can be rendered hydrophobic (water repellent), potentially with greater efficiency and less cost. This is generally accomplished with a class of synthetic chemicals called organo-silanes (OS) (Daniels et al. 2009) or with natural materials such as Tung Oil (Lin et al. 2021). Contact angle measurements are often used as indicators of water repellency. Soil contact angles greater than 90° are considered hydrophobic. Still greater contact angles can be more desirable when super-hydrophobicity is required as well as to account for factors such as non-uniform application and potential degradation. Work by Keatts et al.

(2018) found a trend of increasing apparent contact angle with higher OS dosage rates. Other studies also noted a positive correlation between increasing OS dosages, apparent contact angles and water entry pressures of treated samples (Kim et al. 2011; Keatts et al. 2018; Saulick et al. 2018; Mahedi et al. 2020; Carillo et al. 1999; Wang et al. 2000 and Choi et al. 2016).

One promising application of EWR is the mitigation of freeze-thaw damage in frost susceptible soils. For example, approximately 60% of the roadways in Iowa are granular. Operation and maintenance of these granular roads are estimated to be around $270 million each year (Satvati et al. 2019). Part of this cost is due to the damage resulting from frost action that occurs as the soil subgrade undergoes periods of freezing and thawing. Three factors must be present in order for frost action to occur: (1) the temperature falls below the pore water freezing point, (2) water is available from shallow or perched groundwater, and (3) the soils are of intermediate grain size, i.e., predominantly silt sized. Control of any of these three factors will prevent the associated damage. Local temperature control is possible through insulation, but this is generally not feasible on a large scale. Using soil that is not frost susceptible is also an option for smaller, new construction projects but is not reasonable for an area as large as the entire Iowa granular road network. It is also not a practical solution for remediation of the existing infrastructure. However, the final variable of controlling water availability is a viable alternative. When freezing soils do not have an external source of water, the frost action is limited to the effect of the freezing pore water. This greatly limits the extent of any potential ice lens formation as well as subsequent frost boils compared to soils which have an external water source. By using OS treatment to create a barrier to infiltration, the potential for frost action can be greatly reduced. There is very little data on the effect of OS on frost-susceptible soils. The objective of this paper is to provide data and analysis on the soil water characteristic curve, apparent contact angle, and water entry head of four frost susceptible soils.

MATERIALS AND METHODS

Four different frost susceptible soils were identified and collected. Soils were collected from the following counties in Iowa where unbound, unsurfaced roads are utilized and experience frost heaving regularly: Buena Vista (BV), Clinton (CC), Keokuk (KK), and Pottawattamie (PC), as shown in Figure 1.

Figure 1. Iowa soil collection locations.

Material Characterization

Each of the four soil samples was fully characterized according to standard methodology outlined by the American Standard Testing Methods (ASTM) with duplicate testing. A summary of the material classification and relevant index properties can be found in Table 1. Grain size distributions were determined for each of the soils using sieve analyses and hydrometer testing. Gradation for the coarse fraction of the samples was determined using the sieve analysis method outlined in ASTM D6913 (ASTM 2017). Testing procedures followed the companion measurement method outlined in ASTM D7928-17 (ASTM 2017). Further classification of the fine material was achieved using determination of the Atterberg limit states. The liquid limit, plastic limit, and plasticity index of each soil were determined according to ASTM D4318 (ASTM 2017). The multipoint liquid limit method (Method A) was used for all soils tested. The plastic limit was determined using the hand-rolling method detailed in ASTM D4318 (ASTM 2017).

According to the Unified Soil Classification System (USCS), BV is classified as a high plasticity clay (CH) while the other three samples are classified as lean clays (CL). The soils are all primarily fine-grained with large portions of silt-sized grains as shown in the grain size distribution curves in Figure 2. According to the US Army Corps of Engineers (1986) frost design soil classification system, the soils tested are classified as F4 frost action soils. This designation is the highest possible classification, and the soils are expected to have a medium to very high degree of frost susceptibility (FHWA 2006). Standard Proctor compaction testing was conducted to identify the soil's maximum dry density and optimum moisture content. Testing procedures followed those outlined by Method A in ASTM D698 (ASTM 2012). The maximum dry density and optimum moisture content was then determined from the peak of the curves.

Table 1. Summary of Iowa Soil Index Properties and Classifications

Parameter	BV	CC	KK	PC
Specific Gravity	2.71	2.64	2.72	2.77
Liquid Limit (%)	62	49.4	43	35.4
Plasticity Index (%)	32.8	26.2	24.6	12.1
Optimum Moisture Content (%)	26.8	19.5	17.5	16.8
Max Dry Unit Weight (kN/m^3)	13.8	16.0	16.7	16.4
USCS Classification	CH	CL	CL	CL
AASHTO Classification	A-7-6	A-7-6	A-7-6	A-6

Soil Water Characteristic Curves (SWCC)

The SWCC were determined using a combination of two testing techniques. The dewpoint potentiometer method was performed using the WP4C Dewpoint Potentiometer from METER Group (METER Group, Inc., Pullman WA) for higher values of suction. This was combined with data recorded from the HYPROP 2 from METER Group for lower levels of suction. Samples used for each test method were compacted using standard Proctor energy at their optimum moisture content to achieve the maximum dry density. Samples were then extruded into the appropriate cylindrical sample holder (12.7 cm height by 5 cm diameter for HYPROP and 1 cm height by 4 cm diameter for WP4C).

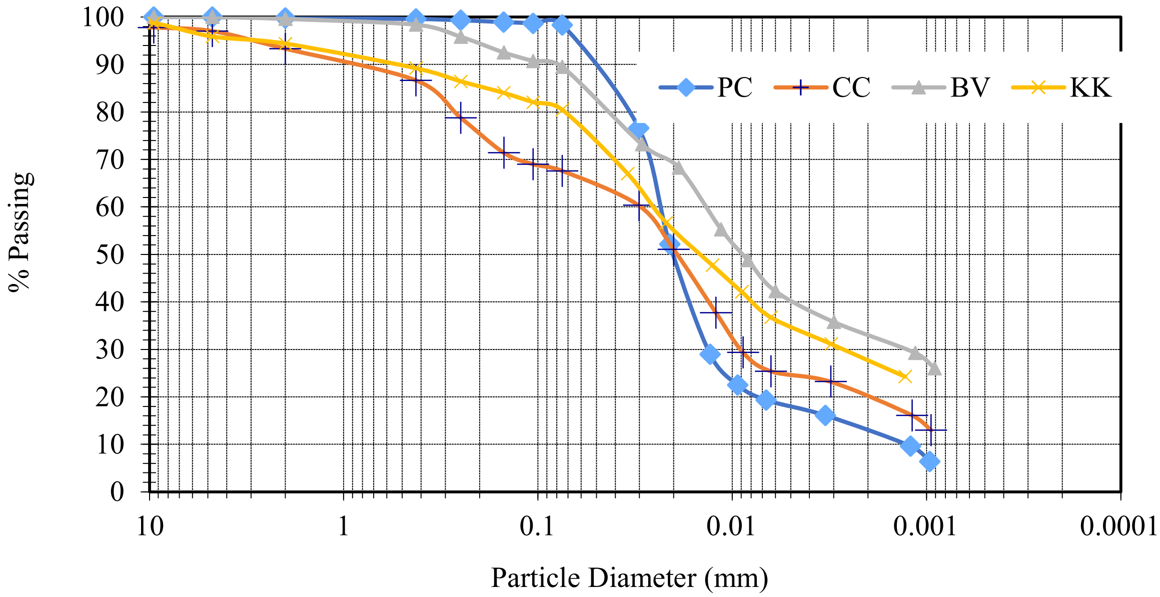

Figure 2. Grain Size Distributions of Iowa Soils.

The WP4C is used for the range of 1MPa to 300MPa which falls on the drier end of the SWCC (METER 2021). The dewpoint potentiometer tests at varying water contents and measures the total suction using the vapor pressure of the soil at its given moisture content. For the WP4C tests, testing methodology followed that described in Dumenu et al. (2017). The specimens were saturated for 24 hours prior to testing. After the mass of the saturated sample was recorded, initial suction readings were taken. After testing, samples were allowed to dry under ambient lab conditions for 24 hours before testing again. The WP4C was calibrated every 24 hours using a 0.5M potassium chloride solution provided by METER Group. For total suction less than 2 MPa, the continuous reading mode was used for one hour. For suctions ranging from 2 MPa to 40 MPa, the precise mode was used. Beyond 40 MPa, the fast mode was used. The mass of the samples was recorded prior to each test. Once the mass of the samples reached a plateau, the testing was terminated. Sample cups were placed in the oven for dry mass determination.

The accuracy of the WP4C is limited in the lower ranges of suction (METER 2021; Leong et al. 2003). Leong et al. (2003) found that total suction measured by the chilled-mirror dew-point technique was consistently greater than the sum of the matric and osmotic suctions when measured independently. The HYPROP makes use of dual tensiometers located at varying heights in the sample to directly measure total suction as the soil dries. Testing and equipment preparation followed the procedures outlined in the HYPROP manual (METER Group, 2018). Samples were saturated in deaired and deionized water for 24-72 hours until water was noticed on the top surface of the soil. HYPROP measurements were taken at least until both tensiometers had reached their cavitation point at which time the test was terminated. The SWCC curves were generated using the accompanying HYPROP-Fit software by METER Group. Data from the WP4C testing was also included in this model. Unsaturated hydraulic conductivity curves were also developed as part of these readings. Pore size (radii) distributions were determined according to methods presented by Lu and Likos (2004).

Chemical Treatment

Two separate OS products were used to test for hydrophobicity: Zydex Terrasil and SIL-ACT ATS-100. Zydex Terrasil (Zydex Industries, Gorwa, India) is a viscous water-soluble liquid that is marketed towards soil modification of secondary road subgrades. SIL-ACT ATS-100 (Advanced Chemical Technologies, Oklahoma City, Oklahoma) is a clear liquid with a density of 0.92 g/cm^3. ATS-100 is approved for use in bridge deck treatment by multiple departments of transportation across the U.S. The Zydex chemical was diluted with tap water at a ratio of 1:10 (Daniels et al. 2009) while the ATS-100 was used as provided by the manufacturer. The process of treating soil samples with OS varied slightly depending on the type of OS used. Soil was treated with the chemicals at a ratio of 1 g OS solution to 2 g of soil (ATS-100) and 1 g of OS solution to 1 g soil for Zydex, in part because of the different consistency. In both cases there was sufficient solution to ensure particle coverage as evidenced by the formation of slurry within the tumbler used for mixing. This allowed for conservation of the chemical while still ensuring saturation of the soil particles.

A 50 g sample of oven dried soil was obtained and placed into an HDPE bottle. The desired amount of OS treatment was then added to the bottle and the two were mixed for 1 minute by shaking vigorously by hand. After shaking, the bottles were placed in a lab tumbler and allowed to continually rotate for 24 hours. The soil / OS mixture was then evenly divided into two weighing dishes. One dish was placed under the fume hood under 'air dried' conditions; the second dish was placed in a 60° oven. Each sample was allowed to dry for 24 hours prior to contact angle testing. After drying, a mortar and rubber-tipped pestle were used to break up any agglomerations of the OS treated soil.

Contact Angle Testing

The general procedure followed the technique developed by Bachmann et al. (2000). The OS-treated soil sample was sprinkled over a piece of double-sided adhesive affixed to a glass microscope slide. Using a soil spatula, light pressure was applied to the coated tape for approximately 10 s. The sides of the glass slide were then tapped to remove any excess soil. This process of applying soil was repeated a minimum of 3 times for each slide to ensure full coverage of the tape. Duplicate slides were prepared for each soil at each of the corresponding OS treatments and drying methods. Contact angle measurements were recorded using the sessile drop technique developed by Bachmann et al. (2000). The prepared slides were placed in a goniometer fixed to a 1.3M USB digital microscope (AVEN model #26700-200, Ann Arbor, Michigan) along with a backlighting source and adjustable sample holding table (Keatts et al. 2018). Contact angle measurements were taken at the three-phase contact line where the horizontal plane of the soil contacted the water droplet. Readings were taken at successively larger water droplets until the horizontal limits of the microscope were reached. A total of 10 measurements for both the left and right sides of the droplet were taken for each droplet size.

Water Entry Head

Water entry head tests were performed with OS from Zydex and not from ATS-100. Soil that was mixed using the OS solution as the molding moisture was compacted into a conventional permeameter cell (Humboldt model HM-5802, Raleigh, NC). The soil was compacted approximately to the dry density as determined by the standard compaction of the treated material used for infiltration testing, not the untreated maximum dry density. The heights of compacted soil in the permeameters were kept relatively constant between 55-60 mm. After compacting, the top filter paper and porous stone were installed, and the soil was allowed to 'dry' within the permeameters with the caps removed and all valves open for 24 hours. After this time, the cell was connected to a standpipe capable of applying positive pressure to the bottom of the sample. Positive water head was then slowly applied to the sample like the methodology used in Keatts et al. (2018). Water head was applied at a rate of approximately 5 cm / min to account for the expected slow infiltration rate. After water was added, the standpipe was observed for slow decreases in height for at least one minute. The critical water entry head was noted when the water in the standpipe failed to reach a stable height, or visual infiltration to the specimen was noticed, or both.

RESULTS AND DISCUSSION

For the SWCC results, curves were fitted using a nonlinear van Genuchten model built into the HYPROP-Fit software. All SWCC data was recorded at the soil's maximum dry density which was achieved through standard Proctor compaction. Figure 3 shows the combined data for all four soils, as well as the resulting pore size distribution. The water retention curves of the four soils are comparable and overlap at the dry end of the curve. The greatest variance in the samples arise from their saturation water content. This is a function of each soil's pore size distribution. The HYPROP measures the initial portion of the SWCC as the soil is just beginning to dry. During this period, larger pores release water first while smaller pores retain water until higher levels of total suction are reached. Compared to the other three soils, BV has a higher volumetric water content and retains its moisture for higher levels of total suction. This is reasonable, however, considering the higher plasticity of the soil. All curves show a gradual release of moisture as would be expected for these fine-grained materials.

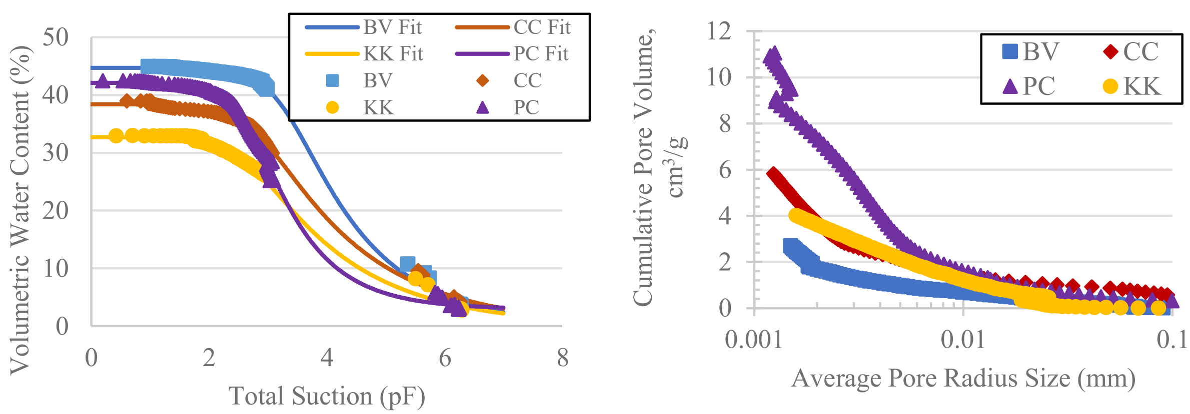

Figure 3. Soil water characteristic curves (left) and pore size distribution (right).

Results for the contact angle testing are shown in Figure 4. All soils were rendered hydrophobic with contact angles at or above 120º. Air dried samples tended to have higher contact angles than their oven dried counterparts. While oven drying is not practical for field application, the two methods were included to gather data on any mechanistic differences. For all chemicals and dosages tested, some variance was noticed between the drying methods, but neither consistently outperformed the other.

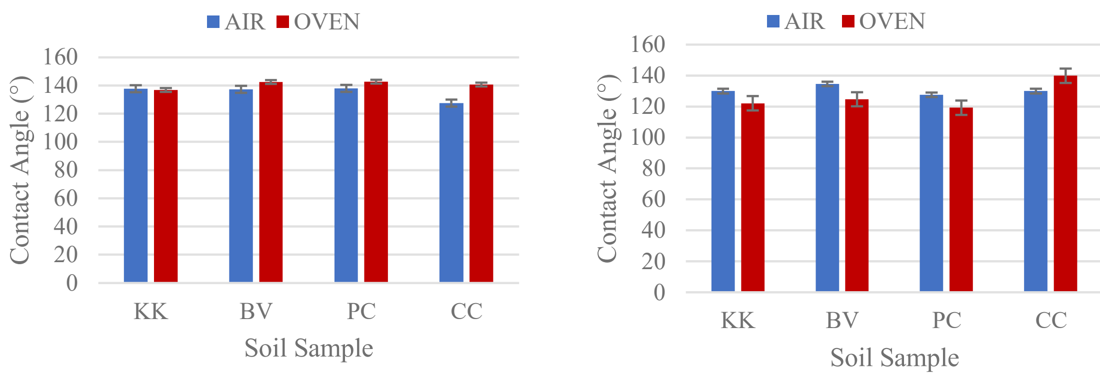

Figure 4. Contact Angle Results for Zydex (Left) and ATS-100 (Right).

Water entry head testing provides more insight into in situ water repellency as it accounts for parameters such as dry density, pore radius, and true contact angle (Keatts et al. 2018). Positive water entry heads were observed for all soils. The water entry head for the soils ranged from 15 cm to 63 cm. Higher water entry head is related to higher contact angles although the lower water entry heads observed in this study are potentially a result of incomplete drying of the treated soils within the permeameter. Drying is an important part of the OS treatment process because it allows the OS molecules to partition to the soil particles as the water evaporates. The authors expect this will be a focus of future testing to ensure adequate drying methods (analogous to curing for lime or cement modification) are implemented in the field. An inverse trend was also noticed with average pore radius and observed breakthrough pressures. According to Bartell et al. (1948), to obtain a fabric of high-water resistance, there must exist not only a high degree of water repellency, but also the pore radii must be small. This trend was also observed in this study (Figure 5).

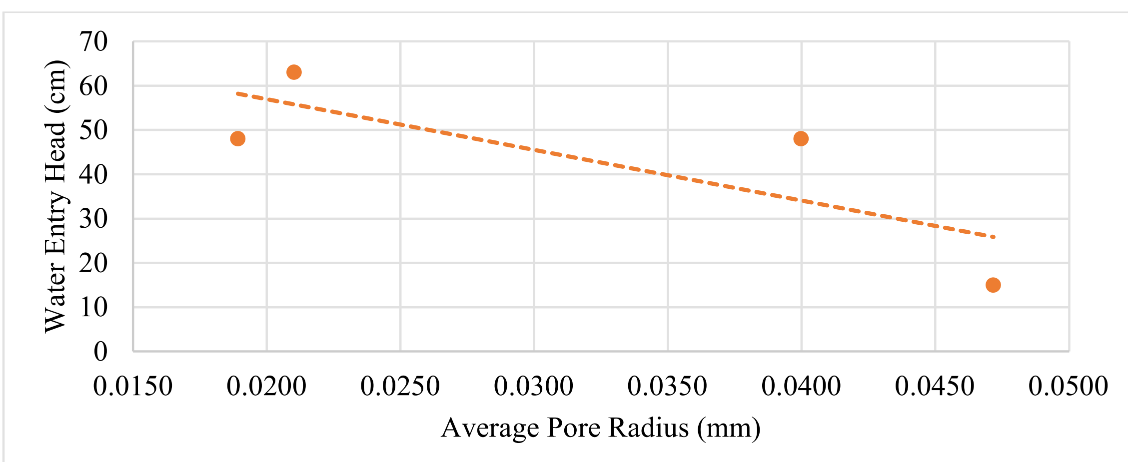

Figure 5. Measured Water Entry Head as a Function of Average Pore Radii.

SUMMARY AND CONCLUSIONS

The objective of this study was to determine the index properties and soil water characteristic curve data of four frost susceptible Iowa soils and to evaluate the extent to which such soils can be rendered water repellent. Effective water repellency was measured using apparent contact angle testing as well as water entry heads. ATS-100 displayed the highest contact angle results (average = 138º for ATS-100 as compared to 129º for Zydex). At a dilution of 1:10, Zydex Terrasil was determined to be a moderately effective treatment for inducing water repellency according to contact angle and water entry head tests. An average contact angle of 129° was observed along with water entry heads ranging from 15 cm to 63 cm. The results of this study suggest that for field application, the in-situ water content is likely to influence the effective dilution.

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