

## International Journal of Geotechnical Engineering



ISSN: (Print) (Online) Journal homepage: www.tandfonline.com/journals/yjge20

# Optimization of water repellency in soils for geotechnical applications

M. Uduebor, J. Daniels, Y. Saulick, W. Naqvi & Bora Cetin

**To cite this article:** M. Uduebor, J. Daniels, Y. Saulick, W. Naqvi & Bora Cetin (2023) Optimization of water repellency in soils for geotechnical applications, International Journal of Geotechnical Engineering, 17:7-10, 753-763, DOI: 10.1080/19386362.2023.2295689

To link to this article: <a href="https://doi.org/10.1080/19386362.2023.2295689">https://doi.org/10.1080/19386362.2023.2295689</a>

	Published online: 30 Dec 2023.
	Submit your article to this journal 🗷
<u>lılıl</u>	Article views: 163
Q <sup>L</sup>	View related articles 🗷
CrossMark	View Crossmark data ௴





### Optimization of water repellency in soils for geotechnical applications

M. Uduebor (ba, J. Danielsa, Y. Saulick (ba, W. Naqvi (bb and Bora Cetin (bb)

<sup>a</sup>Civil and Environmental Engineering, The University of North Carolina at Charlotte, 9201 University City Blvd, Charlotte, NC, USA; <sup>b</sup>Department of Civil and Environmental Engineering, Michigan State University, East Lansing, MI, USA

#### **ABSTRACT**

Applying organo-silanes (OS) as water-repellent additives can enhance soil properties, crucial for use as moisture barriers in infrastructures like roads, landfills, and tunnels. This study used four soil samples and glass beads, treated with three OS products at dosages from 1:1 to 1:1000. Laboratory tests included contact angle, water drop penetration, and breakthrough pressure on 216 samples. Results showed increased hydrophobicity with higher OS dosages, with contact angles over 110° and Water Drop Penetration Test times above 3600s. However, effectiveness plateaued at certain dosages, indicated by electrical conductivity and pH changes. The primary factors (94.6% influence) were soil type, OS product, dosage, and drying condition, while reaction time, and leaching/washing had a minor impact (5.4%). Treated soils could sustain a hydrostatic head of up to 17 kPa. These insights aid in optimizing water-repellency treatments for soil performance and infrastructure durability in geotechnical applications.

#### ARTICLE HISTORY

Received 31 July 2023 Accepted 5 December 2023

#### **KEYWORDS**

Hydrophobic; contact angle; breakthrough pressure; grain size; water-repellency

#### 1. Introduction

Moisture changes result in significant stress on all elements of the pavement system, resulting in the need for maintenance or failure. The problem is further exacerbated when these changes in moisture conditions are seasonal and become even more problematic in roads built on poor subgrade material like frost-susceptible soils, which under suitable conditions keep absorbing moisture under the influence of suction forces resulting in frost heaving (Daniels et al. 2021; Mahedi et al. 2020; Uduebor, Adeyanju, et al. 2022). Repeated frost heaving and thaw weakening within these soils result in damage, leading to annual recurrent maintenance expenditure, road closures, weight restrictions, poor riding experience, and other economic impacts (Brooks et al. 2022; Uduebor, Daniels, et al. 2022; Wasif Naqvi et al. 2022). Seasonal freezing and thawing can contribute up to 75% of pavement degradation (Dore et al. 2005; Yuan, Che, and Tang 2021), and it is estimated that over 2 billion is spent annually on pavement maintenance and restoration due to frost action in the US (FHWA,1999).

Traditional frost mitigation techniques focus on controlling either one or more of the three basic requirements for frost heaving; 1. The presence of frost-susceptible soils (FSS) (silt-sized fractions), which are soils that promote the migration of water towards a freezing front resulting in the formation of an ice lens. 2. Sub-freezing temperatures result in the freezing of water within the soil pores (Daniels et al. 2021; Uduebor, Daniels, et al. 2022). Methods employed include increasing pavement thickness when designing with such soils (usually considering reduced strength due to moisture weakening and frost action), replacing with more suitable backfill material, preventing/intercepting water by use of barrier, and drainage systems (low and/or high permeability soils, geosynthetics) and modifying such soils using

lime and/or cement (de Jesús Arrieta Baldovino, dos Santos Izzo, and Rose 2021). While such methods have been majorly successful, they result in significant labour, time, and resource costs.

Water repellency has been recently explored for use in civil and geotechnical engineering where it can find utility in engineering construction, particularly where removing, resisting, and retaining water is required for the stability and safety of civil infrastructure (Brooks et al. 2022; Mahedi et al. 2020; Uduebor, Adeyanju, et al. 2023). Barrier systems prevent the infiltration of water into areas where it is undesirable (landfill sites, road pavement foundations, tunnels, etc.), and engineered water repellency (EWR) can be a solution.

EWR is a technique for imparting water-repellent properties to soils and is an innovative method for mitigating moisture migration and frost action in road pavements. Soils can be artificially made water-repellent by treating them with water-repellent additives called organo-silanes (OS), which form a covalent, irreversible bond with silica and metal-based substrates, a major component of soil. The modification is permanent as the bond that binds the organic functional groups (R) is the same siloxane (Si-O-Si) bond found in other minerals such as silicon dioxide.

This approach of direct soil modification follows efforts by (Lambe 1951; Lambe, Kaplar, and Lambie 1969), which indicated that four types of water-repellent chemicals yielded a reduction in heave for Boston Blue clay, New Hampshire silt, and Fort Belvoir sandy clay. Several studies carried out using OS such as Dimethyldichlorosilane (DMDCS) and trichloro(octadecyl)silane (OTS), polydimethylsiloxane (PDMS) is available in the literature, and water-repellent chemistry (Choi et al. 2016; Debano 2015; Lin et al. 2019; Lourenço et al. 2018). Organosilanes come in diverse types and have benefits suitable for particular use cases. Water



soluble OS can be mixed with water used to mold and compact soils. This is particularly important when ensuring treatment is carried out effectively and is compatible with current road pavement construction methods. OS that can be utilized directly is more effective for topical spray applications, where only a thin top layer is required to be hydrophobic. They have the advantage of direct utilization and not requiring 'activation' before use.

Other materials used to impart soil hydrophobic properties include Tung oil, Linseed oil (Lin et al. 2019), and wax (Bardet et al. 2015). Advancements in OS and water-repellent additives manufacturing have led to their availability at lower costs, safe application, and use, e.g. as used in food applications (Chorianopoulos et al., 2017; Gkana et al., 2017). Recent formulations which are water-soluble mixtures allow for concentration dilution and effective treatment when applied at the surface or molded with soils during compaction (Daniels and Hourani 2009) with the bonding reaction and hydrophobicity developing as the soil dries.

To successfully establish engineered water repellency as a means for moisture control and frost heave mitigation by designers and engineers, there is a need to develop treatment specifications, obtain optimal OS dosage concentrations, and explore the effects of varying treatment conditions (drying, reaction time, leaching/washing) on treatment outcome. This paper explores EWR in frost susceptible soils and examines the influence of treatment variables on its optimization in frost susceptible soils. It also establishes baseline criteria for using water repellency in geotechnical applications.

#### 2. Materials and methodology

#### 2.1 Soil

Natural soils and glass beads were utilized in this study for testing and analysis. Four soils were collected from distinct locations in the US; Fairbanks in Alaska (AK-FB), Pottawatomie County in Iowa (IA-PC), Asheville in North Carolina (NC-AS), and Hanover silt (NH-HS) from the US Army Cold Regions Research and Engineering Laboratory (CRREL) in New Hampshire. Samples received were air dried and prepared for testing and analysis. Glass beads (Soda Lime, type S) of grain sizes ranging from 0.05 mm (about 0 in) to 1.85 mm (about 0.07 in) were mixed in

proportion to model an average of all the four soil samples given.

#### 2.1.1 Material characterization

Index property and other tests were performed according to the standard ASTM procedures (ASTM 2017a, D4318; ASTM 2023, D854; ASTM 2021, D7928; ASTM 2021, D698; ASTM 2017b, D6913). A summary of the index properties, material classifications, and frost susceptibility classification from the U.S. Army Corps of Engineers (U.S. Army Corps of Engineers 1965) is given in Table 1.

#### 2.2 Organosilane selection

While several products abound to impart hydrophobic properties to materials, three (3) organosilane chemicals were selected for this study based on ease of use, environmental considerations, and cost. The organosilanes were broadly grouped into two categories; (i) 'water-soluble,' requiring dilutions in water to achieve water repellency (through a process of hydrolysis), and (ii) 'use-as-is' which do not require any additional mixing and can be directly mixed in with soil.

#### 2.2.1 Water-soluble OS products

DOWSIL™ IE 6683 (OS1) is a water-based silane/siloxane emulsion that can be used as supplied or diluted further in water for water-repellency treatment of surfaces. It is particularly suited to porous construction materials and bonds with the substrate to produce a durable hydrophobic treatment.

Terrasil (OS2) from Zydex Industries is a viscous, watersoluble, and reactive soil modifier that permanently modifies the soil surface, making it hydrophobic. OS2 is safe and has been utilized in previous studies as a soil modifier and performance enhancer, particularly in stabilization for pavement applications (Oluyemi-Ayibiowu and Uduebor 2019)

#### 2.2.2 "Use-as-is" OS product

SIL-ACT<sup>®</sup> ATS-100 (OS3) is a clear, durable silane treatment product utilized in masonry, concrete, and stone waterproofing. Treated surfaces become repellent to water, chloride, waterborne contaminants, and weathering elements. They have been utilized by the Departments of Transportation (DOTs) of many states for the treatment of parking decks, bridges, airport

	SOIL				
Soil Property	AK-FB	IA-PC	NC-AS	NH-HS	
Specific Gravity, Gs	2.67	2.74	2.65	2.68	
#4 Sieve (4.75 mm)	97.6	100	89.51	79.8	
#10 Sieve (2mm)	96.0	99.8	73.32	74.18	
#40 Sieve (0.425 mm)	93.4	99.6	67.08	52.69	
#200 Sieve (0.075 mm)	84.5	98.4	30.52	42.41	
Silt content (%) (75µm-2µm)	75.65	86.67	26.47	37.52	
Clay content (%) (< 2µm)	8.87	11.69	4.05	4.88	
Liquid Limit, LL	41.0	33.73	38.44	41.8	
Plastic Limit, PL	NP	NP	NP	NP	
Optimum Moisture Content (%)	20.7	17.5	18.50	10.6	
Max. Dry Unit Weight (kN/m³)	14.1	16.3	15.02	19.5	
USCS Classification	ML	CL	SM/SC	ML	
AASHTO Classification	A-5	A-6	A-4	A-4	
Frost Susceptibility Classification	F4	F3	F3	F4	



pavements, and highways (Behravan et al. 2022; Khanzadeh Moradllo, Sudbrink, and Lev 2016).

#### 2.3 Treatment protocol

Initial treatment was carried out at a dosage concentration of 1:10 (OS: Soil, batched by weight) after Uduebor, Adeyanju, et al. (2023), to determine the relative effectiveness of the products and select the most effective three (two. water-soluble and one use-as-is). While higher concentrations of some products could prove more effective, there is a need to account for the associated costs of the increased volume of material required. Further treatment at varying dosage concentration ratios was carried out to determine optimal dosage concentrations. For the use-as-is product (OS3), the soil and OS were manually mixed for one minute in a 250 ml (8.45 oz) HDPE bottle and set up on a tumbler to react for 24 hours (30 cycles/min). For water-soluble OS (OS1, OS2), the OS product was mixed with DI water to achieve a Liquid/Solid Ratio of 1:1 to ensure maximum coverage of the soil samples. Therefore, for 50 g (0.11lb) of soil at 1:10 dosage, 5 g (0.011lb) of the OS was diluted in DI water to make up 50 g for mixing.

To observe the impact of drying conditions, the resulting mixture was then split into two parts placed into cans, and dried under two different drying conditions: air drying in an air-conditioned laboratory (temperature 22°C, relative humidity ~ 21%) and an electric oven at 60°C. While air-dried samples simulated conditions closest to field results, the ovendried samples gave the maximum possible drying conditions available. Oven-dried samples were dried for 24-48 hours (about 2 days) and then cooled for 24 hours in a desiccator to prevent an enhancement of the water repellency during measurement (Roy et al. 1992).

#### 2.4 Water repellency assessment

#### 2.4.1. Contact angle (CA) test

CA measurement was carried out following protocols by (Feyvisa, Daniels, and Pando 2017) which described a dynamic approach to improve the repeatability of tests carried out on coal fly ash (Bachmann, Ellies, and Hartge 2000). A double-sided adhesive tape was attached to a glass slide and dried samples were sprinkled on the other coated side and compressed for 10s using a 10g weight. The slide was then tapped carefully to remove any excess soil grains, creating a monolayer of soil on the tape surface. This process of application was repeated twice to ensure full coverage of the tape and duplicate slides were also prepared for each test. The soil

specimen was placed on a goniometer (Ramehart Instruments, 260-U1, standard goniometer, #150512) made up of a microscopic camera, along with a fibre optic backlighting source and an adjustable sample holding table (Figure 1). Drops of deionized water were placed on the surface of the specimen utilizing a FlowTrac II (Geocomp Products) in volume increments of 20 µl. The drop was gradually advanced with continual horizontal image capturing and measurements were taken for each drop size. The drop advancement continued until a stable CA was observed, taken as the sample's apparent CA. CA measurements less than 90° are considered wettable/hydrophilic, while angles measured between 90° and 150° are considered hydrophobic. Angles above 150° are taken to be superhydrophobic (King 1981). The observed minimum and maximum are also given as standard deviations from this value.

While laboratory tests provide optimal conditions for the treatment of soils using a different OS, there is a need to investigate the effect of varying treatment variables on the resulting hydrophobicity of engineered soils. A number of variables were considered; Soil type (S) (IA-PC (fine-grained) , NH-HS (coarse-grained), GB (coarse-grained)), Organosilane Product (OS) (OS1, OS2, OS3), Dosage (D) (1:10, 1:50, 1:100), Reaction Time (R) (0.25, 4, 12, 24 hours), Leached Condition (Washed, Unwashed), to determine the effect of changes on the water repellency imparted to the soil sample. A total of 216 samples were tested using a parametric study comprising the various variables and the results were analysed using a twostep analysis of variance (ANOVA) test.

#### 2.4.2 Water drop penetration time (WDPT) test

20 g of dried samples were utilized for the WDPT measurements. The samples were placed in aluminium cans and tapped lightly on the side to get a uniform surface. This is to prevent the rolling of the water droplets after placement. Three drops of deionized water (50  $\pm$  1  $\mu$ L volume) were placed on the soil surface with a pipette. The tests were conducted under a constant temperature of ~ 25°C, and RH of ~ 21% without draft to minimize evaporation during the experiment. Any penetration less than or equal to 1 second was taken as instantaneous. All measurements were terminated after 1 hour (3600s), and WDPTs exceeding 3600s (1 hour) were assigned as extremely water-repellent.

#### 2.4.3 Breakthrough head tests

While CA and WDPT tests indicate the degree of water repellency of soils using a planar surface, they do not give information about the ease with which water can penetrate the pore space between particles. Breakthrough head tests offer a good

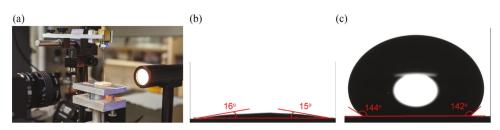


Figure 1. (a) Contact Angle measurement (b) hydrophilic (<90°) (c) hydrophobic (>90°).

correlation for water repellency concerning the treatment and provide more insights into the performance of the treated material under practical use conditions. According to (Carrillo, Yates, and Letey 1999), if the CA is greater than 90° (i.e. hydrophobic), a positive pressure is required to force liquid into the capillary space. The pressure required to force the liquid is referred to as the breakthrough pressure head.

The method established by (Feyyisa et al. 2019) using a flexible wall permeameter setup as described in (ASTM D5084 2016) was adopted. It shares a similar operational concept to the rigid wall permeameter approach reported in previous studies (Carrillo, Yates, and Letey 1999; Fink 1970; Letey, Carrillo, and Pang 2000). It avoids side wall leakage using a confining pressure on a flexible membrane. The breakthrough pressure is identified as the pressure corresponding to the maximum rate of change of the pressure-time data series. This correlates with the results of (Fink and Myers 1969), who identified the breakthrough pressure as the point where a change in the slope of the linear section of the pressure-time series plot occurs.

Oven-dried soil samples (35 mm (1.38 inches) by 70 mm (2.76 inches)) compacted at Optimum Moisture Content (OMC) were mounted on a triaxial cell. A constant cell pressure of 138 kPa was applied using a FlowTrac II system (Geocomp) to prevent preferential flow between the flexible membrane and the soil sample. The soil sample was mounted on a porous stone and the bottom was flushed to remove entrapped air. The input flow line was set up with a pressure transducer (PX409-030GUSBH from Omega Engineering, Inc.) via the inflow valve to determine the pressure applied while the outflow valve was kept open to allow pore air to escape during water infiltration. DI water was supplied at incremental pressures, with successive pressure increments of 1kPa, using another FlowTrac II. Each pressure increment was maintained for 300s and the volume of water passing through the sample at constant pressure was monitored. The pressure and volume response were logged every second using software paired with the pressure transducer. Breakthrough pressure was selected based on the pressure/volume-time series plot. The breakthrough pressure test ended after water penetrated through the sample (indicated by volume change).

Electric Conductivity (EC) and pH measurements were carried out on untreated and treated samples using a Mettler Toledo probe. The dried samples were mixed with Deionized water (~1 μS/cm) at a liquid-to-solid ratio of 2:1 for 24 hours and the supernatant was extracted for testing.

#### 3. Results and discussion

#### 3.1 Contact angle

The results of CA tests are presented in Figure 2 Soils treated have remarkably high CA (>110°) and are all hydrophobic after treatment. An observable result was the effect of drying on the CA results. Air-dried samples had a lower CA compared to oven-dried samples. This is because the water-repellent properties of the soil are affected by dry conditions (Lee et al. 2015). Tests carried out by (Leelamanie, Karube, and Yoshida 2008) also show that CA is reduced with increased humidity. While oven-dried samples provide the maximum possible CA measurement for a given soil sample, air-dried samples provide values closer to what is obtainable under field conditions.

Figure 3 shows the change in CA with respect to dosage concentration. There is a gradual decrease in the CA with decreasing OS concentration. While the overall trend is consistent, in the case of IA and AK soils for OS1, there was more variability between 1:10 and 1:50 concentrations. The OS1 product appears more sensitive to the increased organic content in AK soil and the higher plasticity of IA soil. Other variability in the results can be attributed to the heterogeneity of the natural samples. Treatment with OS2 achieves a higher CA even at lower concentrations (1:1000) due to the high concentration of the active ingredients (65-70%) compared to OS1 (40%). There was an insufficient quantity of OS3 to saturate and treat the soil samples by the 'use as is' product at lower concentration ratios resulting in poor treatment of the soil surface. Results from (Choi et al. 2016) indicate that only approximately 40% of the soil particle surface is required to be treated for measurable hydrophobicity. This means a high CA does not necessarily mean full surface treatment. This characteristic is also observed by the marginal increment in contact angle with increased dosage.

Energy-dispersive X-ray spectroscopy (EDX) analysis performed (See Table 3) shows that the soils possess some quantity of silica which is favoured for silanization (15.48–33.47%). The soils also have a good amount of Iron (5.58–17.57%), except for glass beads. Studies have also shown good adsorption of organosilanes by Iron and Aluminium oxide surfaces, with better adsorption on iron oxide surfaces than aluminium at lower concentrations (Quinton, Thomsen, and Dastoor 1997). At lower dosage concentrations (1:500, 1:1000), there is a marked difference between the CA of different soil samples, with soils (AK-FB, GB) performing better than others. Various products also have different compositions as indicated in Table 2 and this may also be responsible for the variation in results, as products bond differently with soils.

#### 3.1.1 Effect of varying treatment variables on the contact angle of engineered soils

Different variables were investigated – soil type, organosilane product, dosage, and drying condition - and their effect on treatment effectiveness. All interactions between the testing variables were considered and the resulting sum of squares indicated the resulting variance in the CA was obtained. Changes in soil type, OS, and dosage contributed to the variation in the values of the CA (~94.59%). Other considerations including reaction time and leaching condition did not contribute much to the variance in the CA (~5.40%). Drying effects on the CA have been established already and were not considered. There was also a correlation between Soil (p = 4.97e-07 < 0.05), OS (p = 1.52e-09 < 0.05), and Dosage (p = 2.28e-09 < 0.05) with the CA, while there is no correlation between Reaction (p = 0.993 > 0.05) and Washing (p = 0.143 > 0.05). In terms of field application, this means, there is no significant need to pause operations to 'cure' or allow for treatment.

The variation in the CA results due to soil type can be explained by the differences in their mineralogical

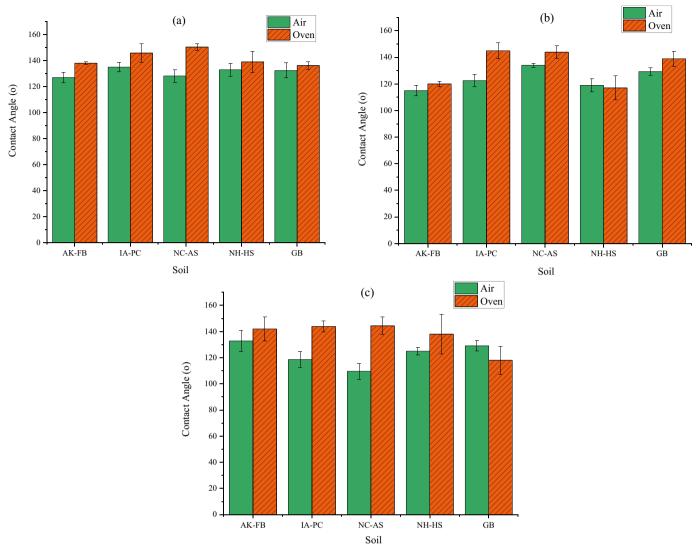


Figure 2. (a-c) Contact angles of soils under two drying conditions after treatment (1:10, OS:Soil, g/g) with (a) OS1 (b) OS2 (c) OS3.

composition which affects the available ions and pH of the resulting mixture. The varying oxide compositions in their respective proportions allow for preferential bonding with the silane-forming siloxane (-Si-O-Si) and -Si-O-Metal bonds. In addition, the available surface area for treatment makes the treatment of finer-grained soils more effective. Studies carried out by (Saulick et al. 2018) have shown that particle size, shape, and roughness can affect the CA of treated soils. OS and dosage effects can be explained by the difference in composition of the three OS utilized in this study which react and bond differently with material surfaces. Their performance can also be affected by the pH of the soil, while the amount of the active ingredient available based on dosage will affect the CA results up to a limiting value where the soil properties indicated above predominantly affect the resulting CA measurement.

A Tukey test carried out with a 95% confidence level showed a similarity between CA results obtained for NH-HS and GB (p = 0.389 > 0.05) while there was no similarity between those for IA-PC and the other two materials tested (p = 2.225e-04, 7.00e-07 < 0.05) indicating that the glass bead material had similar contact results to the NH-

HS sample. Dosage, Washing, and Reaction Time showed no similarities in the results obtained from their varying test conditions.

#### 3.2 Effect of treatment on chemical properties

#### 3.2.1EC

Electrical conductivity was used to track ionic activities in solution and to establish the excess or decrease of ions following treatment. An increase in EC after treatment relates to excess chemical addition to achieve water repellency. There is a marked change in EC at 1:50 dosage concentration for OS1, 1:100 for OS2, and 1:10 for OS3. EC is also a good indicator of excess ions in solution or a measure of excess OS left in solution after treatment. From Figure 4, EC drops after treatment except for treatment with ZD and XA, which have higher EC values indicating an excess in solution. This trend is not repeated in NC-AS where all treatment EC is higher than untreated soil. This could be indicative of excess OS for all treatments or the difference in pore fluid composition and/or mineralogy. OS2 treatment showed a marked increase in EC results with increasing dosage concentration attributable to the

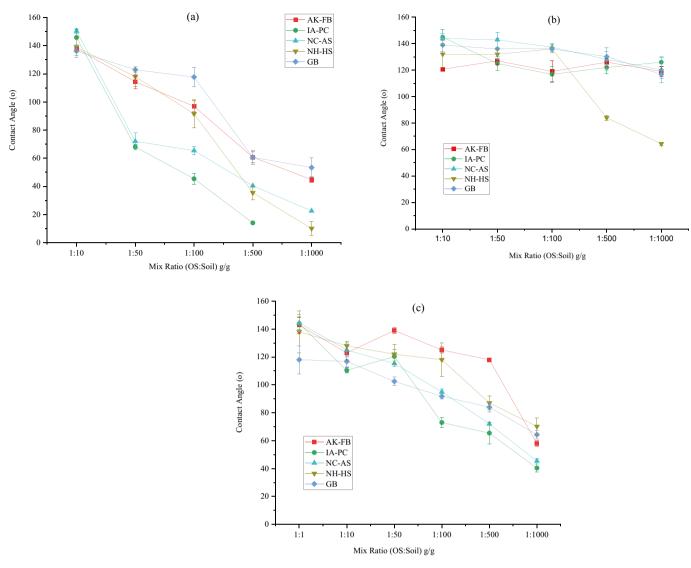


Figure 3. (a-c) Contact angles of soils with varying treatment dosage concentrations (a) OS1 (b) OS2 (c) OS3.

Table 2. Summary of treatment products and active chemicals.

Туре	ID	Product Name	Active compound	Composition in Solution (%)	Color	Specific Gravity (25°C)	рН	Density (g/cm <sup>3)</sup>
Water Soluble	OS1	DOWSIL™ IE 6683	Alkoxysilane, Polydimethylsiloxane	40.0	Milky white	1.0	4.0-6.0	
	OS2	TERRASIL	Alkoxy-Alkylsilyl Compounds	65.0–70			Neutral to acidic	1.01–1.05
Use-as-is	OS3	SIL-ACT® ATS-100	Alkyltrialkoxysilane (Isobutyltrimethyoxysilane)	90–100	Clear	0.92		0.92

Table 3. Elemental composition of soils from EDX analysis.

Table 51 Elemental composition of soils from EBX analysis.							
Element	NC-AS	IA-PC	GB	NHHS	AK-FB		
Carbon (C)	11.44	8.92	21.18	9.63	20.85		
Oxygen (O)	34.89	42.5	41.71	40.98	29.35		
Sodium (Na)	3.87	13.82	8.24	-	-		
Aluminum (Al)	7.6	9.06	0.24	12.23	6.99		
Silicon (Si)	23.89	19.72	24.66	15.48	33.47		
Calcium (Ca)	1.49	1.25	2.05	-	1.35		
Iron (Fe)	16.84	13.82	-	17.57	5.58		
Zinc (Zn)	-	0.73	-	0.96	-		
Magnesium (Mg)	-	2.3	1.93	1.75	1.06		
Potassium (K)	-	1.7	-	1.4	1.35		

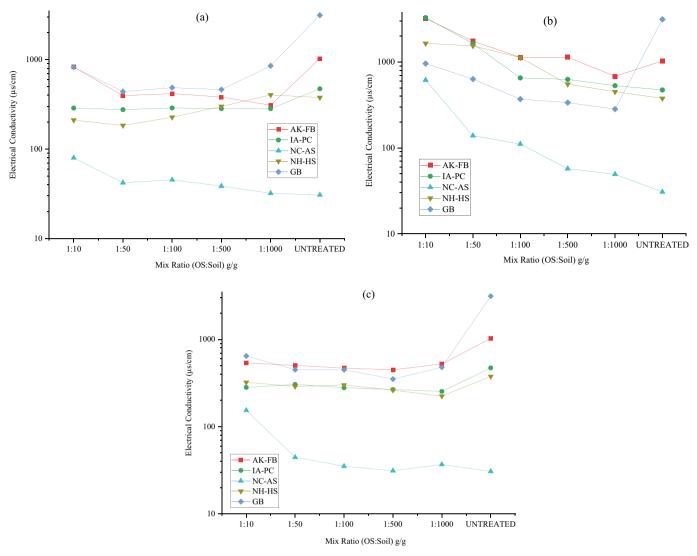


Figure 4. (a-c) electric conductivity of soils with varying dosage concentrations (a) OS1 (b) OS2 (c) OS3.

composition of the OS and resulting reaction products. Increased conductivity could also indicate increased osmotic potential which will result in moisture absorption by the excess salt in the treated soil. This will impact the water-repellent performance of the treated soil and inhibit its ability to serve as a capillary barrier or sustain hydrostatic head.

#### 3.2.2pH

The results of pH tests carried out on treated and untreated samples are presented in Figure 5 There is a good correlation between the EC and pH of treated samples, and both could serve as good indicators for determining optimal treatment. Where there is sufficient utilization of OS, pH remains stable. In cases where the OS is in excess (1:50 for OS1, 1:100 for OS2, and 1:10 for OS3), pH changes based on the composition of the OS to become more acidic. This could be important to note when optimizing treatment in certain applications where the effects of excess OS could impact agricultural land or waterways is a major concern. Variations in results for NCAS treated with OS3 can be attributed to the heterogeneity of samples.

#### 3.3 Water drop penetration time test

The results of the WDPT tests carried out are shown in Figure 6a-c. Some untreated soils (AK-FB, GB) were slightly water repellent with penetration times of 128s (about 2 minutes) and 2s, respectively. The AK-FB sample possesses copious quantities of decayed organic matter and is humic in nature. This results in an apparent hydrophobicity that disappears after mixing. The glass beads are made up of soda lime, which in the amorphous state possesses some form of repellency. For treated samples, there is a marked increase in penetration times with increasing dosage concentration. All samples treated with OS2 were extremely water repellent even at lower concentrations (1:1000). Some studies have developed relationship equations for CA and WDPT (Feyyisa et al. 2019; Keatts et al. 2018), but the models developed cannot be easily transferred across soil samples due to the variations in material and other test conditions as established earlier. At best, it is sufficient to indicate a positive correlation between the two.

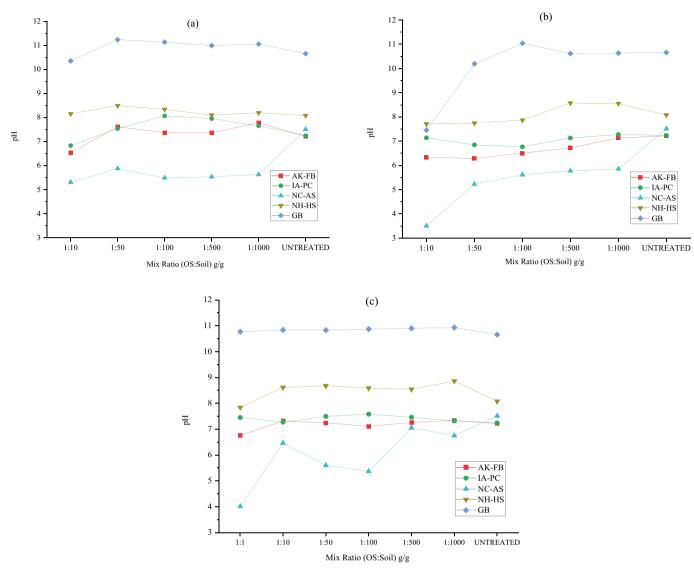


Figure 5. (a-c) pH of soils with varying dosage concentrations (a) OS1 (b) OS2 (c) OS3.

#### 3.4 Breakthrough pressure test

The WDPT does not provide information on the performance of these soils under a pressure head since the water droplet does not impart any considerable pressure on the soil's surface. The breakthrough pressure test provides relevant information useful for engineers in design and construction. Figure 7 shows the water entry (breakthrough pressure) of tested soils under varying dosage concentrations. There is a marked increase in the pressure required to infiltrate the treated soil with dosage. Breakthrough pressures of up to 23 kPa were measured for treated samples.

It can be observed that there was higher breakthrough pressure for the fine-grained sample (IA-PC) even at lower dosages compared to the others. This is because soil properties like grain size affect the results, with fine-grained soils with larger surface areas treatable by the OS and smaller void spaces in the compacted sample. The smaller capillary pore that otherwise would have aided the transport of water through the frost susceptible soil is now moot due to the particle surface being water-repellent. This shows that treatment improves the

permeability properties of frost susceptible soils and makes them suitable for use as capillary barrier materials, preventing the transport of water through them and mitigating the effects of frost action.

#### 4. Conclusion

EWR in soils can be an effective solution for moisture control and improving the water-repellency properties of in-situ soil. This study was carried out to determine the optimal treatment dosage and treatment protocols affecting water repellency in soils. The conclusions of this study are given below.

 Major factors influencing water repellency in soils include soil type, OS (Organosilane) product, dosage, and drying conditions. Other factors like reaction time and leaching do not significantly impact water repellency. For field operations, engineers and designers should consider soil type, OS selection, and dosage as key factors for treatment decisions.

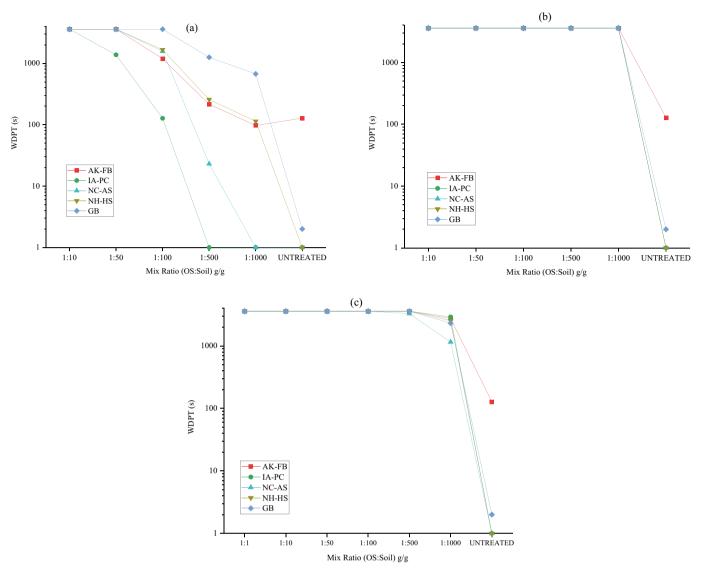


Figure 6. (a-c) water drop penetration times of soils with varying dosage concentrations (a) OS1 (b) OS2 (c) OS3.

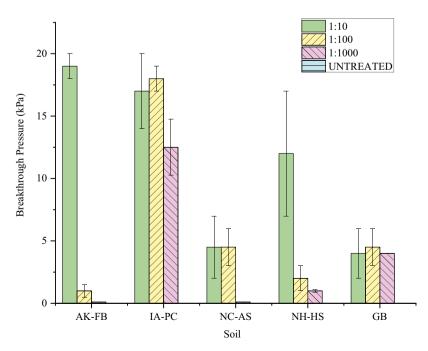


Figure 7. Breakthrough pressure of soils with varying dosage concentrations using OS2. Breakthrough pressures measured for 5 soil materials using OS2 organosilanes at varying dosage concentrations. The results show a decrease in breakthrough pressure followed with decreasing dosage concentration.

- CA test results indicate a positive correlation with dosage concentration, with OS2 treatment proving effective even at lower concentrations (1:1000) and minimal marginal increase in CA measured after 1:50. Due to varying OS types, engineers should specify a target CA to be met rather than a fixed dosage concentration for engineering applications. Optimal dosage concentrations can be preliminarily accessed in the field by monitoring EC and pH changes post-treatment.
- CA and WDPT (Water Drop Penetration Time) are useful but do not provide any information on engineering performance. Breakthrough tests provide some data on the engineering performance of treated soils. OS2-treated soil samples demonstrated increased breakthrough pressures with dosage increases at uniform density, making them suitable for moisture control applications. Finegrained soils exhibit higher breakthrough pressures due to larger surface areas and smaller void spaces.

These results suggest that treated soils can serve as effective barriers or moisture control materials in geotechnical applications. By modifying in-situ soils with EWR treatment frost susceptibility can be eliminated and costly soil replacement can be avoided, saving limited resources. EWR can also be utilized to manage moisture and mitigate shrink-swelling in expansive soils by limiting water transport into the soil matrix.

#### **Acknowledgments**

This work was part of a larger experimental campaign, inclusive of field, laboratory, and numerical aspects as funded by the U.S. National Science Foundation (Award #1928813). Soils were provided by the Iowa Highway Research Board (Keokuk County), the North Carolina Department of Transportation (Ashe County), and the Cold Regions Research and Engineering Laboratory, a U.S. Army Corps of Engineers, Engineer Research and Development Center. The authors would like to appreciate Mya Mitchell, an undergraduate student in the Department of Civil and Environmental Engineering, UNC Charlotte, and Cadet David Ragan, from the U.S. Military Academy in West Point who carried out tests on the project as part of their summer research internship and Advanced Individual Academic Development (AIAD) project, respectively. We would also like to thank the reviewers for taking the necessary time and effort to review the manuscript. We sincerely appreciate all their valuable comments and suggestions, which helped us in improving the quality of the manuscript.

#### **Disclosure statement**

No potential conflict of interest was reported by the author(s).

#### **Funding**

The work was supported by the National Science Foundation [1928813].

#### ORCID

M. Uduebor http://orcid.org/0000-0002-3579-7043 Y. Saulick http://orcid.org/0000-0003-0789-1582 W. Naqvi http://orcid.org/0000-0002-1621-2943 Bora Cetin http://orcid.org/0000-0003-0415-7139

#### **Data availability statement**

The datasets generated during and/or analysed during the current study are available from the corresponding author upon reasonable request.

#### References

- ASTM International. 2016. "D5084 Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter." Astm D5084, 4.
- ASTM International. 2017a. ASTM D4318-17. Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils. https://doi.org/ 10.1520/D7928-21E01.
- ASTM International. 2017b. "D6913: Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis." ASTM D6913 (17). https://doi.org/10.1520/D6913\_D6913M-17
- ASTM International. 2021. ASTM D698: Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort (12 400 Ft-Lbf/ft3 (600 kN-M/m3), ASTM International. https://doi. org/10.1520/D0698-12R21.
- ASTM International. 2021. "Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis." ASTM D7928. https://doi. org/10.1520/D7928-21E01.
- ASTM International. 2023. "ASTM D854 Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer." ASTM D854 ASTM International 4. https://doi.org/10.1520/D0854-23.
- Bachmann, J., A. Ellies, and K. H. Hartge 2000. Development and Application of a New Sessile Drop Contact Angle Method to Assess Soil Water Repellency, www.elsevier.com/locate/jhydrol.
- Bardet, J. P., M. Jesmani, N. Jabbari, and S. D. N. Lourenco. 2015. "Permeability and Compressibility of Wax-Coated Sands." Géotechnique 64 (5): 341-350. https://doi.org/10.1680/Geot.13.P.118. https://doi.org/10.1680/GEOT.13.P.118.
- Behravan, A., S. M. Aqib, N. J. Delatte, M. T. Ley, and A. Rywelski. 2022. "Performance Evaluation of Silane in Concrete Bridge Decks Using Transmission X-Ray Microscopy." International Journal of Offshore and Polar Engineering 12 (5). https://doi.org/10.3390/app12052557.
- Brooks, T., J. L. Daniels, M. Uduebor, B. Cetin, M. Wasif Naqvi, and P. D. Student. 2022. Engineered Water Repellency for Mitigating Frost Action in Iowa Soils 448-456. https://doi.org/10.1061/9780784484012.046.
- Carrillo, M. L. K., S. R. Yates, and J. Letey. 1999. "Measurement of Initial Soil-Water Contact Angle of Water Repellent Soils." Soil Science Society of America Journal 63 (3): 433-436. https://doi.org/10.2136/ SSSAJ1999.03615995006300030002X.
- Choi, Y., H. Choo, T. S. Yun, C. Lee, and W. Lee. 2016. "Engineering Characteristics of Chemically Treated Water-Repellent Kaolin.' Materials 9 (12). https://doi.org/10.3390/ma9120978.
- Chorianopoulos, J., L. Iacumin, A. B. Cabello, E. N. Gkana, A. I. Doulgeraki, N. G. Bautista-Gallego, G.-J. E. Nychas, and A. Garrido-Fernández. 2017. "Anti-Adhesion and Anti-Biofilm Potential of Organosilane Nanoparticles Against Foodborne Pathogens." Frontiers in Microbiology 8. https://doi.org/10.3389/ fmicb.2017.01295.
- Daniels, J. L., and M. S. Hourani 2009. "Soil Improvement with Organo-Silane." U.S.-China Workshop on Ground Improvement Technologies 2009, 217-224. https://doi.org/10.1061/41025(338)23
- Daniels, J. L., W. G. Langley, M. Uduebor, and B. Cetin. 2021. "Engineered Water Repellency for Frost Mitigation: Practical Modeling Considerations." Geo-Extreme 2021:385-391. https://doi. org/10.1061/9780784483701.037.
- Debano, L. F. 2015. "Infiltration, Evaporation, and Water Movement as Related to Water Repellency." Soil Conditioners 155-164. https://doi. org/10.2136/SSSASPECPUB7.C15.
- de Jesús Arrieta Baldovino, J., R. L. dos Santos Izzo, and J. L. Rose. 2021. "Effects of Freeze-Thaw Cycles and Porosity/Cement Index on Durability, Strength and Capillary Rise of a Stabilized Silty Soil Under Optimal

- Compaction Conditions." Geotechnical and Geological Engineering 39 (1): 481-498. https://doi.org/10.1007/s10706-020-01507-y.
- Dore, G., P. Drouin, P. Pierre, and P. Desrochers 2005. Estimation of the Relationships of Road Deterioration to Traffic and Weather in Canada. http://www.bv.transports.gouv.qc.ca/mono/0965375.pdf.
- Feyyisa, J. L., J. L. Daniels, and M. A. Pando. 2017. "Contact Angle Measurements for Use in Specifying Organosilane-Modified Coal Combustion Fly Ash." Journal of Materials in Civil Engineering 29 (9): 29(9. https://doi.org/10.1061/(asce)mt.1943-5533.0001943.
- Feyyisa, J. L., J. L. Daniels, M. A. Pando, and V. O. Ogunro. 2019. "Relationship Between Breakthrough Pressure and Contact Angle for Organo-Silane Treated Coal Fly Ash." Environmental Technology & Innovation 14. https://doi.org/10.1016/j.eti.2019.100332.
- FHWA. 1999. "A Quarter Century of Geotechnical Research" FHWA-RD-98-139. Accessed 23 02, 2023 https://www.fhwa.dot.gov/publications/ research/infrastructure/geotechnical/98139/04.cfm
- Fink, D. H. 1970. "Water Repellency and Infiltration Resistance of Organic-Film-Coated Soils." Soil Science Society of America Journal 34 (2): 189-194. https://doi.org/10.2136/SSSAJ1970. 03615995003400020007X.
- Fink, D. H., and L. E. Myers. 1969. "Synthetic Hydrophobic Soils for Harvesting Precipitation." Symposium on Water-Repellent Soils Riverside (Carlifornia). 221-240.
- Gkana, E., Doulgeraki, A., Chorianopoulos, N, and Nychas, G. J (2017). Anti-adhesion and Anti-biofilm Potential of Organosilane Nanoparticles against Foodborne Pathogens. Frontiers Microbiology. 8. https://doi.org/10.3389/fmicb.2017.01295.
- Keatts, M. I., J. L. Daniels, W. G. Langley, M. A. Pando, and V. O. Ogunro. 2018. "Apparent Contact Angle and Water Entry Head Measurements for Organo-Silane Modified Sand and Coal Fly Ash." Journal of Geotechnical and Geoenvironmental Engineering 144 (6): 144(6. https://doi.org/10.1061/(ASCE)GT.1943-5606.0001887.
- Khanzadeh Moradllo, M., B. Sudbrink, and M. T. Ley. 2016. "Determining the Effective Service Life of Silane Treatments in Concrete Bridge Decks." Construction and Building Materials 116:121-127. https:// doi.org/10.1016/J.CONBUILDMAT.2016.04.132.
- King, P. M. 1981. "Comparison of Methods for Measuring Severity of Water Repellence of Sandy Soils and Assessment of Some Factors That Affect Its Measurement." Australian Journal of Soil Research 19 (3). https://doi.org/10.1071/SR9810275.
- Lambe, T. William. 1951. Soil Testing for Engineers 9. Wiley. https://doi. org/10.2134/agronj1951.00021962004300120015x
- Lambe, T. W., C. W., Kaplar, and T. J., Lambie. 1969. "Effect of Mineralogical Composition of Fines on FrostSUusceptibility of Soils." U S Engr Dept-Cold Regions Research & Eng Laboratory-Tech. (Report
- Leelamanie, D. A. L., J. Karube, and A. Yoshida. 2008. "Characterizing Water Repellency Indices: Contact Angle and Water Drop Penetration Time of Hydrophobized Sand." Soil Science & Plant Nutrition 54 (2). https://doi.org/10.1111/j.1747-0765.2007.00232.x.
- Lee, C., H.-J. Yang, T. S. Yun, Y. Choi, and S. Yang. 2015. "Water-Entry Pressure and Friction Angle in an Artificially Synthesized Water-Repellent Silty Soil." Vadose Zone Journal 14 (4). https://doi. org/10.2136/vzj2014.08.0106.
- Letey, J., M. L. K. Carrillo, and X. P. Pang. 2000. "Approaches to Characterize the Degree of Water Repellency." Canadian Journal of

- Fisheries and Aquatic Sciences 231-232:61-65. https://doi.org/10.1016/ S0022-1694(00)00183-9.
- Lin, H., S. D. N. Lourenço, T. Yao, Z. Zhou, A. T. Yeung, P. D. Hallett, G. I. Paton, K. Shih, B. C. H. Hau, and J. Cheuk. 2019. "Imparting Water Repellency in Completely Decomposed Granite with Tung Oil. Journal of Cleaner Production 230:1316-1328. https://doi.org/10.1016/ J.JCLEPRO.2019.05.032.
- Lourenço, S. D. N., Y. Saulick, S. Zheng, H. Kang, D. Liu, H. Lin, and T. Yao. 2018. "Soil Wettability in Ground Engineering: Fundamentals, Methods, and Applications". Acta Geotechnica 13(1). Springer Verlag. https://doi.org/10.1007/s11440-017-0570-0.
- Mahedi, M., S. Satvati, B. Cetin, and J. L. Daniels. 2020. "Chemically Induced Water Repellency and the Freeze-Thaw Durability of Soils.' Journal of Cold Regions Engineering 34 (3): 04020017. https://doi.org/ 10.1061/(ASCE)CR.1943-5495.0000223.
- Oluvemi-Avibiowu, B. D., and M. A. Uduebor. 2019. "Effect of Compactive Effort on Compaction Characteristics of Lateritic Soil Stabilized with Terrasil." Journal of Multidisciplinary Engineering Science Studies 5 (2): 2458-2925. www.jmess.org.
- Quinton, J., L. Thomsen, and P. Dastoor. 1997. "Adsorption of Organosilanes on Iron and Aluminium Oxide Surfaces." Surface and Interface Analysis 25 (12): 931–936. https://doi.org/10.1002/(SICI)1096-9918(199711) 25:12<931:AID-SIA325>3.0.CO;2-F.
- Roy, W. R. \*., I. G. \*. Krapac, S.-F. J. \*. Chou, and R. A. Griffin. 1992. "Technical Resource Document: Batch-Type Procedures for Estimating Soil Adsorption of Chemicals." Report (Issue 87)
- Saulick, Y., S. D. N. Lourenço, B. A. Baudet, S. K. Woche, and J. Bachmann. 2018. "Physical Properties Controlling Water Repellency in Synthesized Granular Solids." European Journal of Soil Science 69 (4): 698-709. https://doi.org/10.1111/EJSS.12555.
- Uduebor, M., E. Adeyanju, Y. Saulick, J. Daniels, and B. Cetin 2022. "A Review of Innovative Frost Heave Mitigation Techniques for Road Pavements." International Conference on Transportation and Development 2022. https://doi.org/10.1061/9780784484357
- Uduebor, M., E. Adevanju, Y. Saulick, J. Daniels, and B. Cetin. 2023. "Engineered Water Repellency for Moisture Control in Airport Pavement Soils." Airfield and Highway Pavements 2023:92-102. https://doi.org/10.1061/9780784484906.009.
- Uduebor, M., J. Daniels, D. Adeyanju, F. Sadiq, and B. Cetin, 2023. "Engineered water Repellency for Resilient and Sustainable Pavement Systems." International Journal of Geotechnical Engineering 17 (5): 530-540. https://doi.org/10.1080/19386362.2023.2241280.
- Uduebor, M., J. Daniels, N. Mohammad, and B. Cetin. 2022. Engineered Water Repellency in Frost Susceptible Soils 457-466. https://doi.org/10. 1061/9780784484012.047.
- U.S. Army Corps of Engineers. 1965. "Soils and Geology- Pavement Design for Frost Conditions." Department of the Army Courses Department of Civil Engineering, University of New Technical Manual TM 5-818-2.
- Wasif Naqvi, M., M. F. Sadiq, B. Cetin, M. Uduebor, and J. Daniels. 2022. "Investigating the Frost Action in Soils." Geo-Congress 2022:257-267. https://doi.org/10.1061/9780784484067.027.
- Yuan, G., A. Che, and H. Tang. 2021. "Evaluation of Soil Damage Degree Under Freeze-Thaw Cycles Through Electrical Measurements.' Engineering Geology 293:106297. https://doi.org/10.1016/J.ENGGEO. 2021.106297.