

Optimization of Water Repellency in Soils for Geotechnical Applications

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ABSTRACT

Water repellency in soils can be achieved by applying water-repellent additives known as organosilanes (OS). This technique enhances soil properties, making them suitable for use as moisture barriers in various infrastructures like road pavements, landfills, and tunnels. To explore the potential of this approach, four soil samples and glass beads were treated with three OS products at varying dosages (ranging from 1:1 to 1:1000, batched by weight). The study included tests such as Contact Angle, Water Drop Penetration, and Breakthrough Head (Pressure). A comprehensive parametric study encompassing multiple variables was conducted on a total of 216 samples. The results demonstrated that increasing OS treatment led to higher hydrophobicity, with contact angles exceeding 110°. However, this trend plateaued at a specific dosage concentration, evident from changes in electrical conductivity and pH measurements, which are practical indicators for field implementation. The primary factors influencing treatment efficacy (approximately 94.6%) were identified as soil type, organosilane product, dosage, and drying condition. Conversely, variables such as reaction time, and leaching/washing, accounted for a minor contribution of approximately 5.4% to the variance in the results. Moreover, Breakthrough Head tests on treated soils exhibited their ability to sustain a head of up to 17 kPa, a significant improvement compared to immediate infiltration in untreated samples. These findings provide essential criteria and specifications for effectively implementing water-repellency treatment in frost susceptible soils, particularly for capillary barriers in geotechnical applications. By understanding these influences, practitioners can optimize water-repellency treatments for enhanced soil performance and long-term infrastructure durability.

Keywords

Hydrophobic, Contact Angle, Breakthrough Pressure, Grain Size

1.0 Introduction

Moisture changes result in significant stress and strain 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 act like sponges and keep absorbing moisture under the influence of suction forces resulting in frost heaving . Repeated frost heaving and thaw weakening 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 et al., 2022; Wasif et al., 2022). Seasonal freezing and thawing can contribute up to 75% of pavement degradation (Dore et al., 2005; Yuan et al., 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, 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 (Baldovino et al., 2021). While such methods have been majorly successful, they result in significant labor, 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., 2022; Uduebor et al., 2023). Barrier systems largely 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. Figure 1 shows the bonding between OS and a sand particle's surface (substrate). 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.

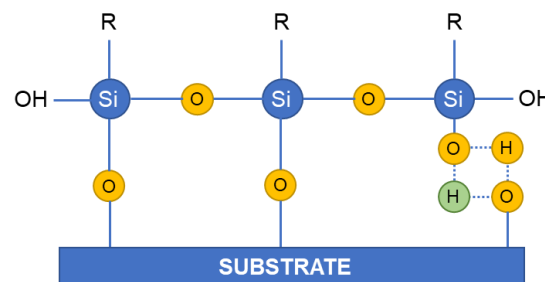


Figure 1: Silane reaction bond with soil surface (Substrate)

This approach of direct soil modification follows efforts by (Lambe, 1951; Lambe et al., 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 various 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 very 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 (Bautista-Gallego 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 & 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.

2.0 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 different 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 U.S. 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 to 1.85 mm 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 D4318; ASTM D854; ASTM D7928; ASTM D698; ASTM D6913). A summary of the index properties, material classifications, and frost susceptibility classification from the US Army Corps of Engineers (U.S. Army Corps of Engineers, 1965) is given in Table 1.

Table 1. Summary of soil index properties and classifications

Soil Property	SOIL			
	AK-FB	IA-PC	NC-AS	NH-HS
Specific Gravity, G _s	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/m ³)	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

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, water-soluble, 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 & Uduebor, 2019)

2.2.2 *“Use-as-is” OS product*

SIL-ACT® ATS-100 (OS3) is a clear, durable silane treatment product utilized in masonry, concrete, and stone waterproofing. Treated surfaces become repellent to water, chloride, and waterborne contaminants as well as weathering elements. They have been utilized by the

Departments of Transportation (DOTs) of many states for the treatment of parking decks, bridges, airport pavements, and highways (Behravan et al., 2022).

Table 2: Summary of treatment products and active chemicals

Type	ID	Product Name	Active compound	Composition in Solution (%)	Color	Specific Gravity (@25°C)	pH	Density (g/cm ³)
Water Soluble	S1	DOWSIL™ IE 6683	Alkoxysilane, Polydimethylsiloxane	40.0	Milky white	1.0	4.0 - 6.0	
	S2	TERRASIL	Alkoxy-Alkylsilyl Compounds	65.0 -70			Neutral to acidic	1.01 – 1.05
Use-as-is	S3	SIL-ACT® ATS-100	Alkyltrialkoxysilane (Isobutyltrimethoxy silane)	90 – 100	Clear	0.92		0.92

2.3 Treatment protocol

Initial treatment was carried out at a dosage concentration of 1:10 (OS: Soil, batched by weight), 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, costs for shipping larger quantities, as well as handling them, make them less cost-efficient. 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 250ml 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 50g of soil at 1:10 dosage, 5g of the OS was diluted in DI water to make up 50g 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 oven-dried samples gave the maximum possible drying conditions available. Oven-dried samples were allowed to dry for 24 - 48 hours 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

Contact angle measurement was carried out following protocols by (Feyyisa et al., 2017) which described a dynamic approach to improve the repeatability of tests carried out on coal fly ash after (Bachmann et al., 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 fiber optic backlighting source and an adjustable sample holding table. Drops of deionized water were placed on the surface of the specimen utilizing a FlowTrac II (Geocomp Products) in volume increments of 20µl. The drop was gradually advanced with continual horizontal image capturing and measurements were taken for each drop size. The drop advancement was continued until a stable contact angle is observed which is taken as the apparent contact angle of the sample. Contact angle 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.

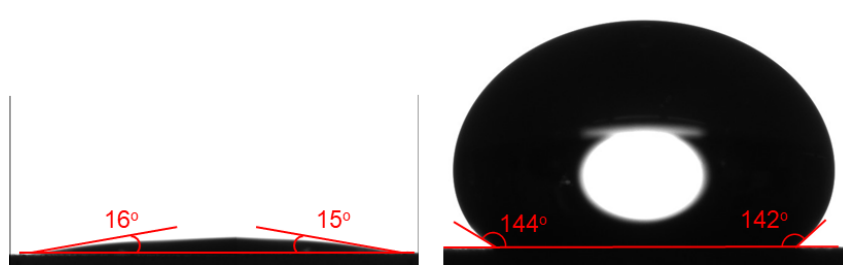


Figure 2: (a) Contact Angle measurement (b) Hydrophilic ($<90^\circ$) (c) Hydrophobic ($>90^\circ$)

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 analyzed using a two-step analysis of variance (ANOVA) test.

2.4.2 Water Drop Penetration Time (WDPT) Test

20g of dried samples were utilized for the WDPT measurements. The samples were placed in aluminum cans and tapped lightly on the side to get a fairly uniform surface. This is to prevent the rolling of the water droplets after placement. Three drops of deionized water ($50 \pm 1 \mu\text{L}$ volume) were placed on the soil surface with a pipette. The tests were conducted under a constant temperature of $\sim 25^\circ\text{C}$, and RH of $\sim 21\%$ 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 were assigned as extremely water-repellent.

2.4.3 Breakthrough Head Tests

While Contact Angle and WDPT tests give an indication of the degree of water repellency of soils using a planar surface, they do not give any information about the ease with which water can penetrate the pore space between particles. Breakthrough head tests offer a good 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 et al., 1999), if the contact angle 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 was adopted. It shares a similar operational concept as the rigid wall permeameter approach reported in previous studies (Carrillo et al., 1999; Fink, 1970; Letey et al., 2000) and 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 & 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 cm by 70 cm) 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 a period of 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 ($\sim 1\mu\text{S}/\text{cm}$) at a liquid-to-solid ratio of 2:1 for 24 hours and the supernatant was extracted for testing.

3.0 Results and Discussion

3.1 Contact Angle

The results of contact angle tests are presented in Figure 3. Soils treated have very high contact angles ($>110^\circ$) and are all hydrophobic after treatment. An observable result was the effect of drying on the contact angle results. Air-dried samples had a lower contact angle compared to sample oven-dried samples. This is because the water-repellent properties of the soil are affected

by dry conditions (Lee et al., 2015). While oven-dried samples provide the maximum possible contact angle measurement for a given soil sample, air-dried samples provide what is obtainable under field conditions.

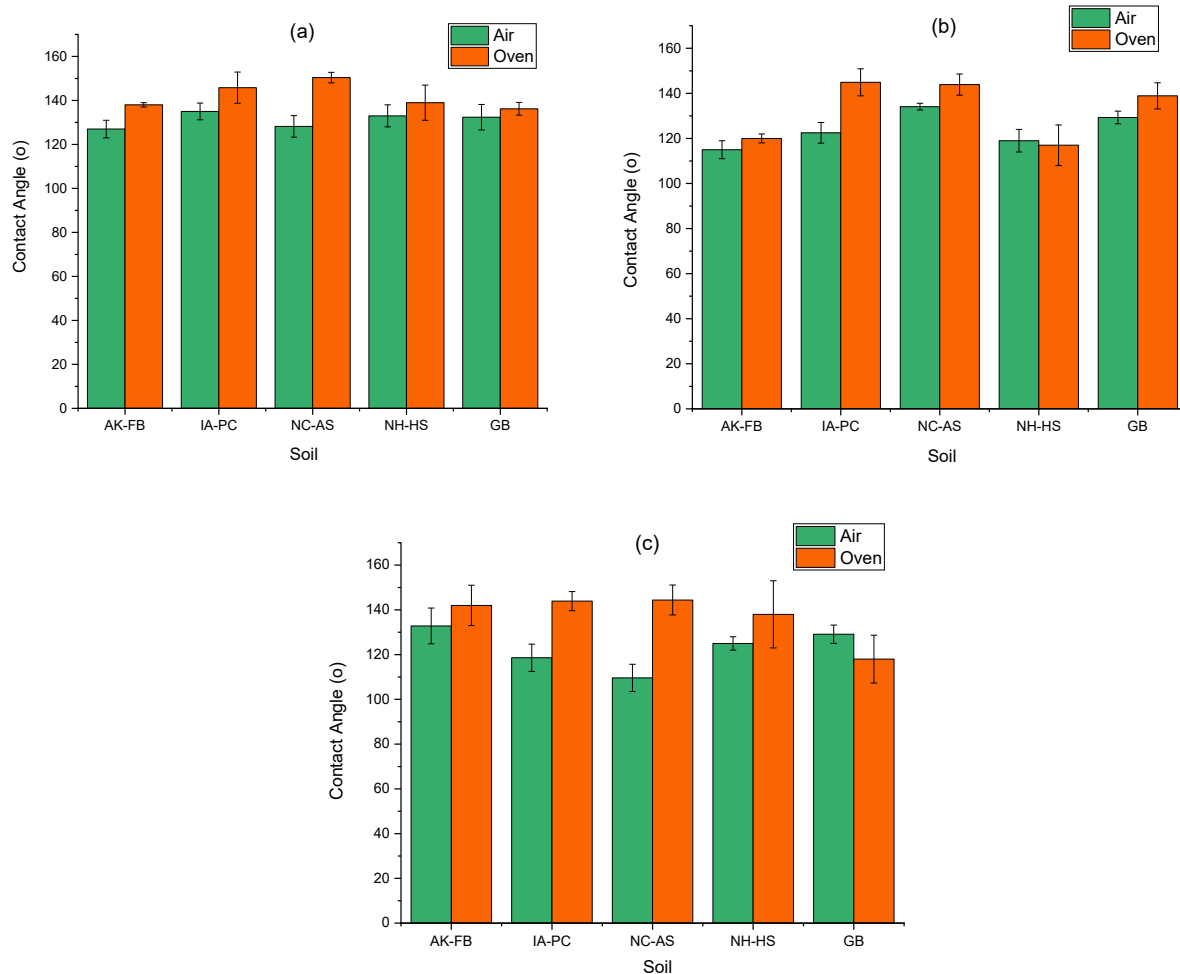


Figure 3a-c: Contact Angles of soils under two drying conditions after treatment (1:10, OS:Soil, g/g) with (a) OS1 (b) OS2 (c) OS3

Figure 4 shows the change in contact angle concerning dosage concentration. There is a gradual decrease in the contact angle with decreasing OS concentration. 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 contact angle does not necessarily mean full surface treatment. This characteristic is also observed by the marginal increment in contact angle with increased dosage.

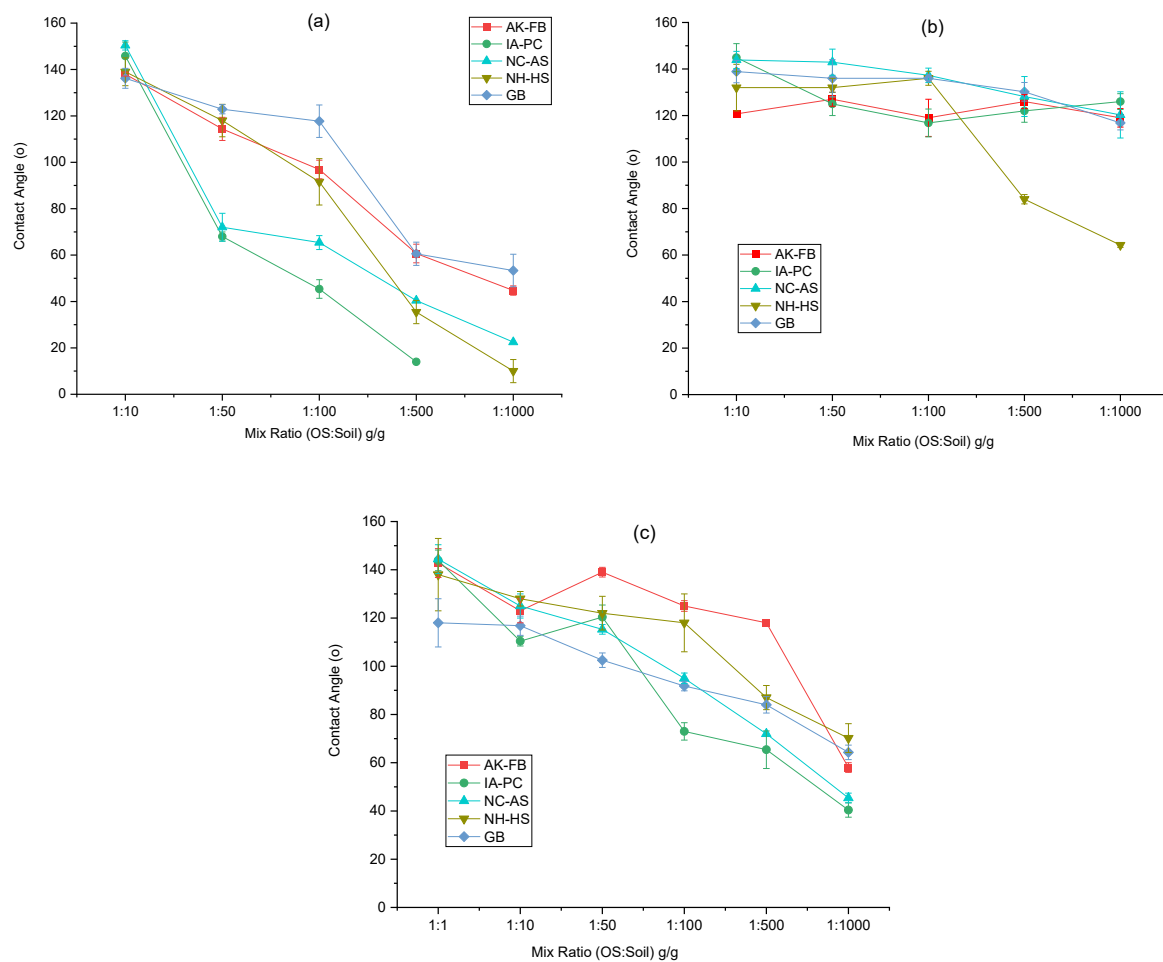


Figure 4a-c: Contact Angles of soils with varying treatment dosage concentrations (a) OS1 (b) OS2 (c) OS3

Energy-dispersive X-ray spectroscopy (EDX) analysis performed (See Table 3) shows that the soils possess some quantity of silica which is favored 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 Aluminum oxide surfaces, with better adsorption on iron oxide surfaces than aluminum at lower concentrations (Quinton et al., 1997). At lower dosage concentrations (1:500, 1:1000), there is a marked difference between the contact angles of different soil samples, with soils (AK-FB, GB) performing better than others. Different products also have different compositions as indicated in Table 2 and this may also be responsible for the variation in results, as different products bond differently with soils.

Table 3: Elemental composition of soils from EDX 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

3.1.1 Effect of Varying Treatment Variables on the Contact Angle of Engineered Soils

Different variables - soil type, organosilane product, dosage, and drying condition - and their effect on treatment effectiveness were investigated. All possible interactions between the testing variables were considered and the resulting sum of squares indicated the resulting variance in the contact angle was obtained. The resulting percentage of each combination as part of the total variance was then plotted into a pie chart (See Fig 6). It can be observed that there is a variation in

the values of the contact angle with changes in soil type, OS, and dosage (a total of 94.59%). Other considerations including reaction time and leaching condition did not contribute much to the variance in the contact angle (5.40%). Drying effects on the contact angle have been established already and were not considered. There was also a correlation between Soil ($p = 4.97\text{e-}07 < 0.05$), OS ($p = 1.52\text{e-}09 < 0.05$), and Dosage ($p = 2.28\text{e-}09 < 0.05$) with the contact angle, while there is no correlation between Reaction Time ($p = 0.993 > 0.05$) 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 contact angle results due to soil type can be explained by the differences in their mineralogical 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 contact angle 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 contact angle results up to a limiting value where the soil properties indicated above predominantly affect the resulting contact angle measurement.

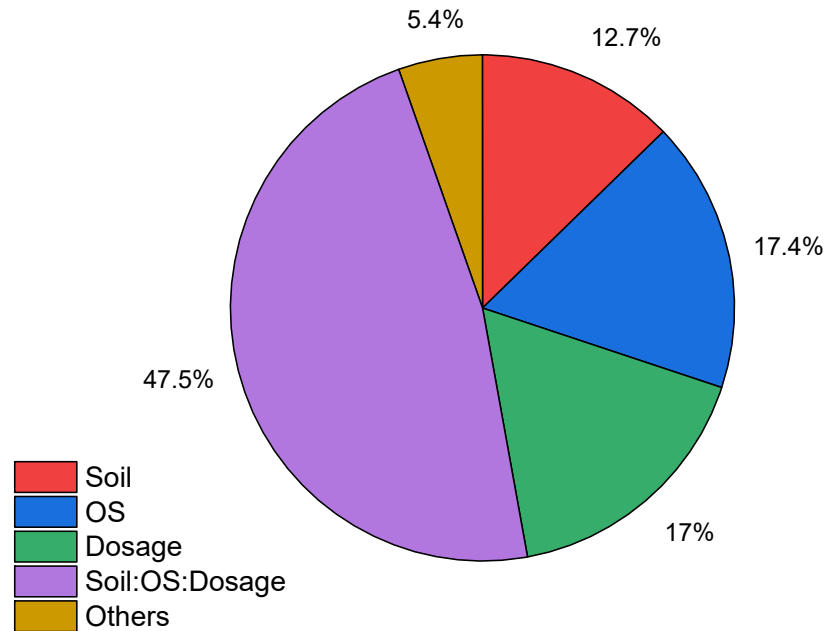


Figure 6: Percentage contribution of variables and to the variance in Contact Angle Results

A Tukey test carried out with a 95% confidence level showed a similarity between contact angle 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

3.2.1 EC

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 6, 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 material composition. 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.

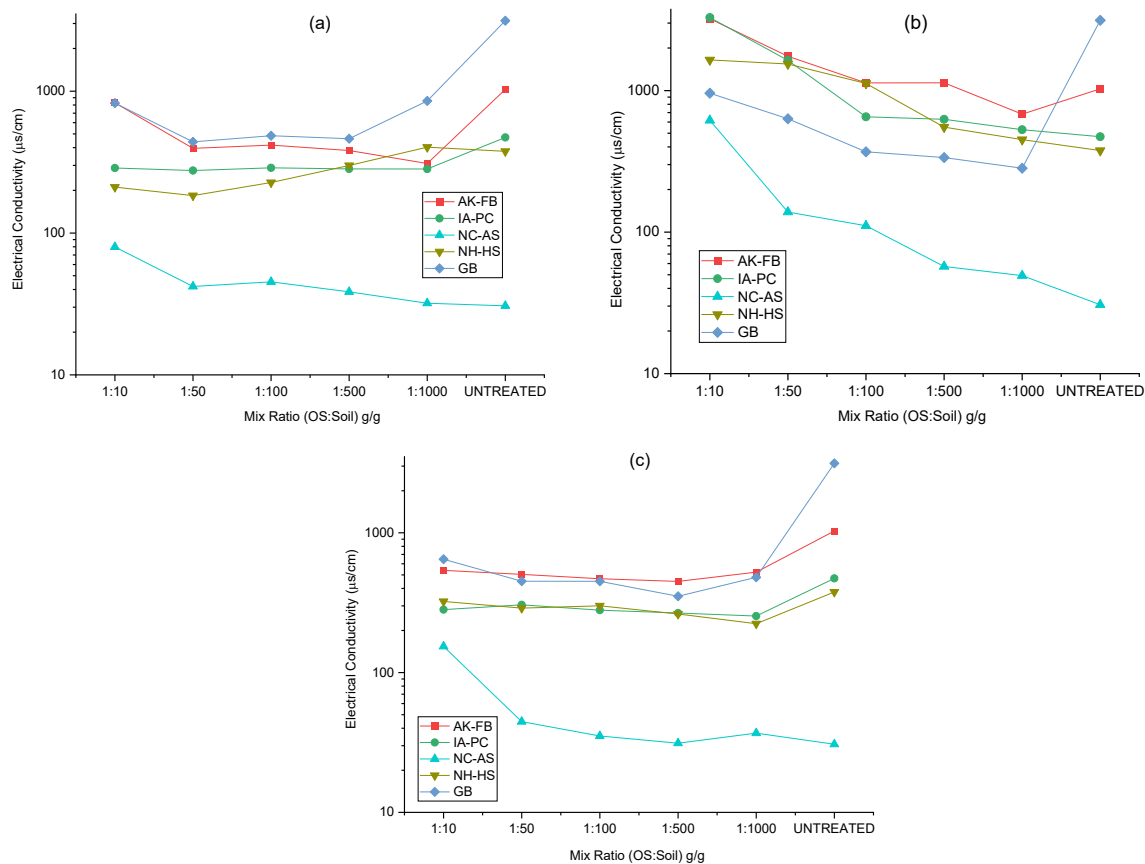


Figure 6a-c: Electric Conductivity of soils with varying dosage concentrations (a) OS1
(b) OS2 (c) OS3

3.2.2 pH

The results of pH tests carried out on treated and untreated samples are presented in Figure 7. 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.

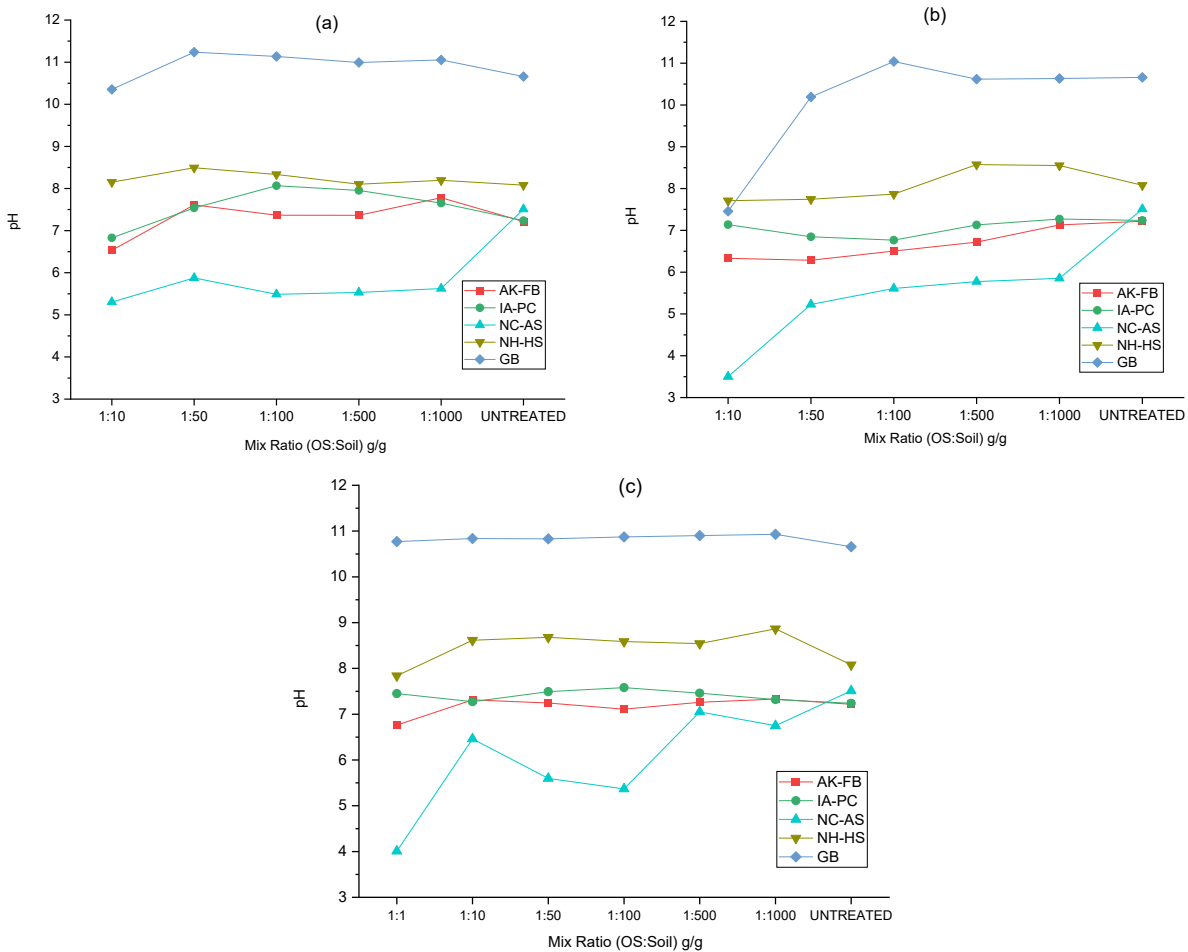
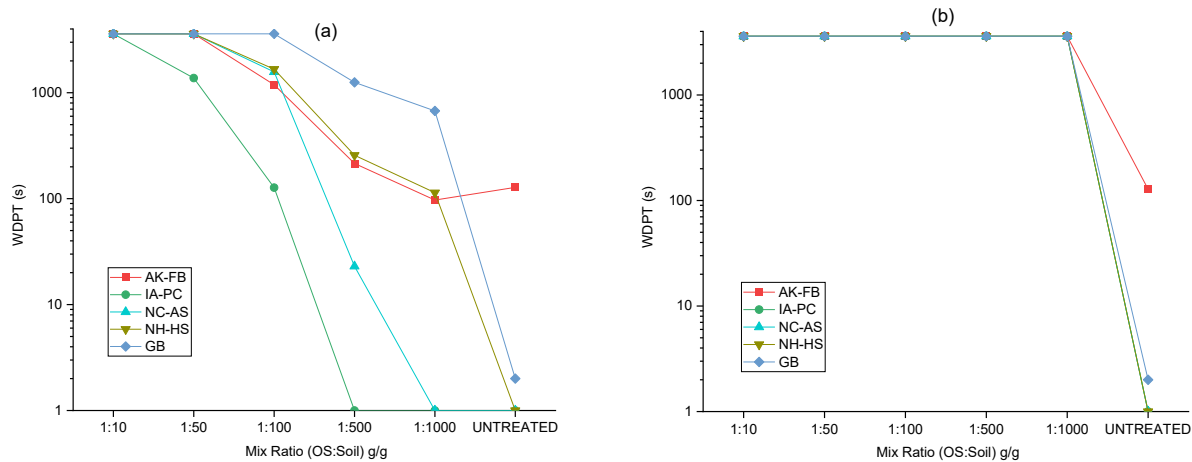


Figure 7a-c: pH of soils with varying dosage concentrations (a) OS1 (b) OS2 (c) OS3

3.3 Water Drop Penetration Time Test

The results of the WDPT tests carried out are shown in Figure 8a-c. Some untreated soils (AK-FB, GB) were slightly water repellent with penetration times of 128s and 2s respectively. The AK-FB sample possesses large 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). There is a good correlation between the Contact Angle results and the WDPT as shown in figure 8d. Some studies have developed relationship equations for contact angle 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 that there is a positive correlation between the two.



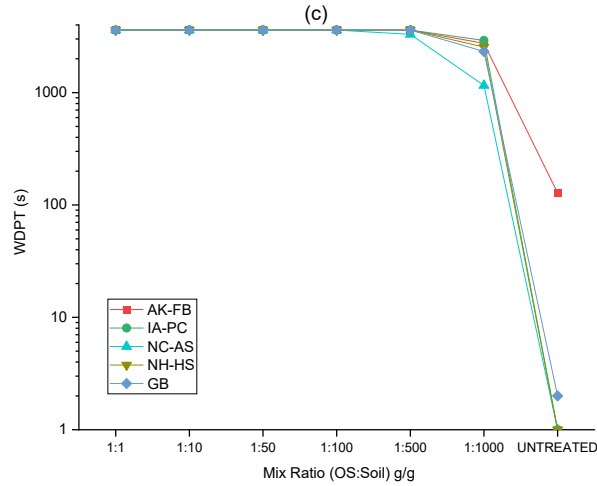


Figure 8a-c: Water Drop Penetration Times of soils with varying dosage concentrations

(a) OS1 (b) OS2 (c) OS3

3.4 Breakthrough Pressure Test

The WDPT does not provide any information on the performance of these soils under a pressure head since the water droplet does not impart any considerable pressure on the surface of the soil. The breakthrough pressure test provides relevant information useful for engineers in design and construction. Figure 9 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.

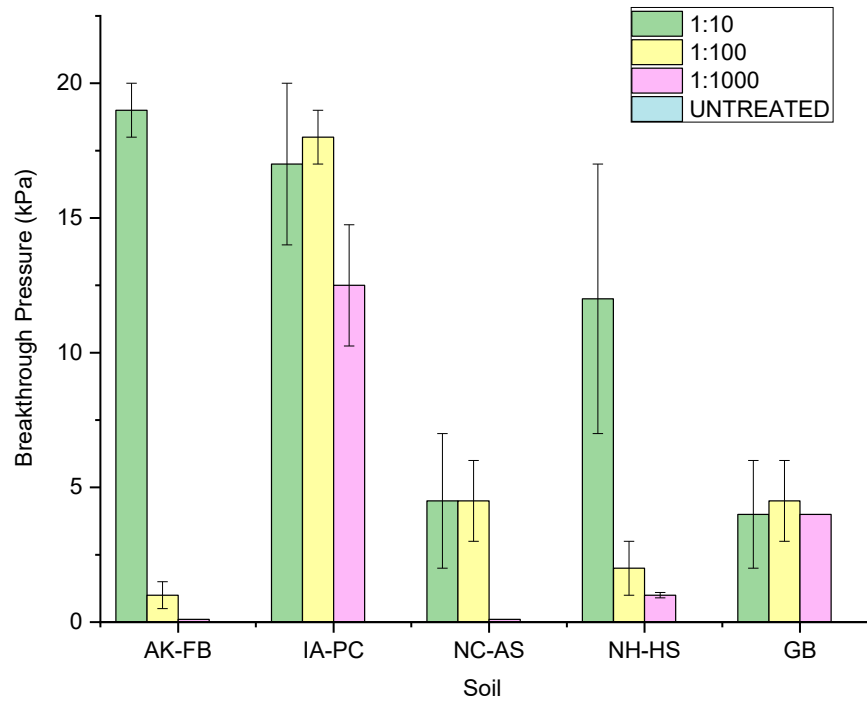


Figure 9: Breakthrough Pressure of soils with varying dosage concentrations using OS2

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 possessing larger surface areas treatable by the OS and also smaller void spaces within the compacted sample. The smaller capillary pore that otherwise would have aided the transport of water through the frost susceptible soil is now shut down 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 - a useful solution for several civil engineering applications. In this study, it has been observed that soil type, OS product, dosage as well as drying condition affect the resulting water repellency imparted to soils. Reaction time and leaching do not have any impact on water repellency. These are important things to consider when considering treatment, particularly in the field. Engineers and designers can focus on the soil type, OS used, and dosage - variables that mostly affect the treatment. Mineralogical analysis carried out on soils revealed that samples contained silica (Si, 15.48 - 33.47%) and Iron (Fe, 5.58 - 17.57%), which bond with the silane forming siloxane (-Si-O-Si) and -Si-O-Metal bonds, improving the water repellency of the soil material. Contact Angle tests provide a good indicator of water repellency in treated samples. There was a correlation between dosage concentration and contact angle, with contact angles of 117° - 150° (hydrophobic - superhydrophobic) measured at 1:10 OS treatment. OS2 treatment had high contact angles even at lower concentrations, making it the most effective OS chemical for use based on the respective treatment dosages selected. Different products impart varying levels of water repellency at different dosages, it is important to specify the target Contact Angle required and not a particular dosage concentration for engineering applications. Electrical conductivity and pH are good indicators for the optimization of treatment. While the OS products and the resulting treated material are safe, it is important to pay attention to resulting changes in EC and pH from the treatment to assess the impact on the environment and for applications where a large change can affect plants or animals. While CA and WDPT are good indicators of water repellency they do not provide any information on the engineering performance of these soils for use as barriers in civil infrastructure applications. The breakthrough head test provides a good indication of the performance of the treated soils for use in moisture control applications and is of

engineering importance for the use of treated soils as capillary barriers in various applications. OS2-treated soil samples sustained heads up to 17kPa. Fine-grained soils possess higher breakthrough pressures due to the larger specific surface area for treatment and the smaller void spaces. Treated soils can be utilized as barrier or moisture control materials for geotechnical applications. This can prove to be an innovative and cost-effective approach to solving frost heave mitigation in road pavements, and foundations where previous techniques have been found cost and labor prohibitive. Instead of stripping soils and replacing them with alternatives, engineers can modify the in-situ soil and render them water-repellent, thereby improving the properties of the frost susceptible material and also saving time and resources. They can also be utilized to manage moisture and mitigate the effect of shrink-swelling in expansive soils by limiting the amount of water transported to them.

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Statements and Declarations

All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

Data availability

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

References

- Astm. (2000). D5084 - Standard Test Methods for Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter. *Astm D5084, 04*(October 1990).
- ASTM. (2012). 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, 3*.
- ASTM D 854. (2002). ASTM D854 Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer. *ASTM International, 04*.
- ASTM D7928. (2021). Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis. *ASTM International, May 2016*.

- ASTM International. (2017a). D6913: Standard Test Methods for Particle-Size Distribution (Gradation) of Soils Using Sieve Analysis. *ASTM International, D6913*(17).
- ASTM International. (2017b). ASTM D4318-17. *Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils*.
- Bachmann, J., Ellies, A., & Hartge, K. H. (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., Jesmani, M., Jabbari, N., & Lourenco, S. D. N. (2015). Permeability and compressibility of wax-coated sands. *Https://Doi.Org/10.1680/Geot.13.P.118*, 64(5), 341–350. <https://doi.org/10.1680/GEOT.13.P.118>
- Bautista-Gallego, J., Iacumin, L., Cabello, A. B., Gkana, E. N., Doulgeraki, A. I., Chorianopoulos, N. G., & Nychas, G.-J. E. (2017). *Anti-adhesion and Anti-biofilm Potential of Organosilane Nanoparticles against Foodborne Pathogens*. <https://doi.org/10.3389/fmicb.2017.01295>
- Behravan, A., Aqib, S. M., Delatte, N. J., Ley, M. T., & Rywelski, A. (2022). Performance Evaluation of Silane in Concrete Bridge Decks Using Transmission X-ray Microscopy. *Applied Sciences (Switzerland)*, 12(5). <https://doi.org/10.3390/app12052557>
- Brooks, T., Asce, S. M., Daniels, J. L., Asce, F., Uduebor, ; Micheal, Cetin, B., Asce, M., Naqvi, M. W., & Student, P. D. (2022). *Engineered Water Repellency for Mitigating Frost Action in Iowa Soils*. 448–456. <https://doi.org/10.1061/9780784484012.046>
- Carrillo, M. L. K., Yates, S. R., & Letey, J. (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., Choo, H., Yun, T. S., Lee, C., & Lee, W. (2016). Engineering characteristics of chemically treated water-repellent Kaolin. *Materials*, 9(12). <https://doi.org/10.3390/ma9120978>
- Daniels, J. L., & Hourani, M. S. (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](https://doi.org/10.1061/41025(338)23)
- Daniels, J. L., Langley, W. G., Uduebor, M., & Cetin, B. (2021). Engineered Water Repellency for Frost Mitigation: Practical Modeling Considerations. *Geo-Extreme 2021*, 385–391. <https://doi.org/10.1061/9780784483701.037-->
- de Jesús Arrieta Baldovino, J., dos Santos Izzo, R. L., & Rose, J. L. (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/FIGURES/12>
- Debano, L. F. (2015). Infiltration, evaporation, and water movement as related to water repellency. *Soil Conditioners*, 155–164. <https://doi.org/10.2136/SSSASPEC PUB7.C15>
- Dore, G., Drouin, P., Pierre, P., & Desrochers, P. (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., Daniels, J. L., & Pando, M. A. (2017). Contact Angle Measurements for Use in Specifying Organosilane-Modified Coal Combustion Fly Ash. *Journal of Materials in Civil Engineering*, 29(9). [https://doi.org/10.1061/\(asce\)mt.1943-5533.0001943](https://doi.org/10.1061/(asce)mt.1943-5533.0001943)

- Feyyisa, J. L., Daniels, J. L., Pando, M. A., & Ogunro, V. O. (2019). Relationship between breakthrough pressure and contact angle for organo-silane treated coal fly ash. *Environmental Technology and Innovation*, 14. <https://doi.org/10.1016/j.eti.2019.100332>
- 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., & Myers, L. E. (1969). *Synthetic Hydrophobic Soils for Harvesting Precipitation*.
- Keatts, M. I., Daniels, J. L., Langley, W. G., Pando, M. A., & Ogunro, V. O. (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). [https://doi.org/10.1061/\(ASCE\)GT.1943-5606.0001887](https://doi.org/10.1061/(ASCE)GT.1943-5606.0001887)
- 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*. Wiley.
- LAMBE TW, KAPLAR CW, & LAMBIE TJ. (1969). EFFECT OF MINERALOGICAL COMPOSITION OF FINES ON FROST SUSCEPTIBILITY OF SOILS. *U S Engr Dept- Cold Regions Research & Eng Laboratory-Tech Report 207*.
- Lee, C., Yang, H.-J., Yun, T. S., Choi, Y., & Yang, S. (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., Carrillo, M. L. K., & Pang, X. P. (2000). Approaches to characterize the degree of water repellency. *Journal of Hydrology*, 231–232. [https://doi.org/10.1016/S0022-1694\(00\)00183-9](https://doi.org/10.1016/S0022-1694(00)00183-9)
- Lin, H., Lourenço, S. D. N., Yao, T., Zhou, Z., Yeung, A. T., Hallett, P. D., Paton, G. I., Shih, K., Hau, B. C. H., & Cheuk, J. (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., Saulick, Y., Zheng, S., Kang, H., Liu, D., Lin, H., & Yao, T. (2018). Soil wettability in ground engineering: fundamentals, methods, and applications. In *Acta Geotechnica* (Vol. 13, Issue 1). Springer Verlag. <https://doi.org/10.1007/s11440-017-0570-0>
- Mahedi, M., Satvati, S., Cetin, B., & Daniels, J. L. (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](https://doi.org/10.1061/(ASCE)CR.1943-5495.0000223)
- Oluyemi-Ayibiowu, B. D., & Uduebor, M. A. (2019). Effect of Compactive Effort on Compaction Characteristics of Lateritic Soil Stabilized With Terrasil. *Journal of Multidisciplinary Engineering Science Studies (JMESS)*, 5(2), 2458–2925. www.jmess.org
- Quinton, J., Thomsen, L., & Dastoor, P. (1997). Adsorption of Organosilanes on Iron and Aluminium Oxide Surfaces. In *SURFACE AND INTERFACE ANALYSIS* (Vol. 25).
- Roy, W. R. *, Krapac, I. G. *, Chou, S.-F. J. *, & Griffin, R. A. (1992). Technical Resource Document: batch-type procedures for estimating soil adsorption of chemicals. In *Report* (Issue 87).

- Saulick, Y., Lourenço, S. D. N., Baudet, B. A., Woche, S. K., & Bachmann, J. (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., Adeyanju, E., Saulick, Y., Daniels, J., & Cetin, B. (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., Adeyanju, E., Saulick, Y., Daniels, J., & Cetin, B. (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., Daniels, J., Naqvi, M. W., & Cetin, B. (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., Sadiq, Md. F., Cetin, B., Uduebor, M., & Daniels, J. (2022). Investigating the Frost Action in Soils. *Geo-Congress 2022*, 257–267. <https://doi.org/10.1061/9780784484067.027-->
- Yuan, G., Che, A., & Tang, H. (2021). Evaluation of soil damage degree under freeze–thaw cycles through electrical measurements. *Engineering Geology*, 293, 106297. <https://doi.org/10.1016/J.ENGGEOL.2021.106297>