Engineered Water Repellency in Frost Susceptible Soils

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ABSTRACT

The presence of frost susceptible soils makes the design, construction, and maintenance of geostructural systems such as pavement difficult. This pronounced in areas that experience seasonally cold weather characterized by freezing sub-zero temperatures. An innovative approach in mitigation is to render frost susceptible soils hydrophobic. This is achieved by treating soils with cost-effective and environmentally compatible polymers in a process that makes them water repellent (Engineered Water Repellency). This paper investigates the treatment of selected frost susceptible soils using commercially available chemicals (ATS-100, Terrasil, DOWSIL IE6683) at varying concentration dosages. The degree of hydrophobicity imparted, and performance was determined using contact angle measurements and Water Drop Penetration Tests. Treatment using ATS-100 and Terrasil were found to be effective even at small concentrations with values ranging from 73° to 144° for ATS-100 and from 103° to 145° for Terrasil. Effective values for DOWSIL IE6683 were obtained at a ratio of 1:10.

INTRODUCTION

The seasonal freezing and thawing of frost susceptible soils (FSS) make them undesirable in construction due to the dramatic changes in thermal and mechanical properties, which have damaging effects on structures constructed on them (Lein et al, 2019). Particularly in road pavements, frost susceptible soils when exposed to moisture and freezing conditions, cause frost heaving (Ahammed, 2018).

One widely accepted definition of FSS, presented by the Highway Research Board Committee on Frost Heave and Frost Action in Soil (1955) is "one in which significant ice segregation will occur when the requisite moisture and freezing conditions are present". The presence of these soils coupled with freezing temperatures and the availability of a water source either within these soils or a high groundwater table provides desirable conditions for frost action. This leads to substantial damage due to large changes in stress, strains, and moisture content from frost heave and freeze-thawing lead to huge recurring maintenance costs estimated at over two billion US dollars annually (FHWA, 1999). Remedial techniques such as stripping and replacement, thermo-siphoning, soil stabilization or increasing pavement thickness are generally cost prohibitive or unfeasible.

Engineered Water Repellency (EWR) is an alternative approach that offers a transformative approach to environmental geotechnology challenges (Debano, 1981; Daniels, 2020) and is currently finding relevance in infiltration control (Daniels et al, 2019), landfill barriers (Subedi et al., 2012) and slope covers (Lourenco et al, 2017) and has proven efficient in the treatment of soils and coal combustion residuals (Feyyisa et al, 2019; Dumenu et al, 2017). Initially, naturally occurring water repellency was first recognized in agricultural and soil science (Schreiner and Shorey, 1910), but with advances in water repellent chemistry and polymer technologies, soil modification has emerged as another application (Daniels et al 2009; Weissenbach 2012). One method of imparting water repellency to soils is through a process known as silanization. This involves treatment of soil with organo-silanes (OS) to form a covalent -Si-O-Si- irreversible bond with silica-based substrate increasing its hydrophobicity (Seed, 2001). A number of studies in EWR using OS chemicals are available in literature (Daniels et al. 2009; Kim et al. 2015; Jordan et al. 2017; Keatts et al. 2018; Saulick et al. 2021).

By treating FSS with cost-effective and environmentally compatible polymers in a process that makes them hydrophobic (water repellent), we can limit the transport of water through these soils, and the process of frost action can be mitigated. This method can prove to be a viable alternative or complement to already existing methods of reducing frost action in soils. Water repellency has also been found to have a significant effect on the retention properties of soils (Beckett, 2016; Dumenu et al, 2017). This paper, as a part of a larger study on the use of Engineered Water Repellency in frost heave mitigation, investigates the treatment of selected frost susceptible soils using commercially available chemicals at varying concentration dosages. The degree of hydrophobicity imparted, and performance was determined with contact angle measurements based on the Sessile Drop Method, Water Drop Penetration Test, and water entry head tests.

MATERIALS AND METHODS

Natural soils were utilized in this study for testing and analysis. Three different FSS were collected from: Boone County in North Carolina (NC-BO), Pottawattamie County in Iowa (IA-PC), and Fairbanks in Alaska (AK-FB). Samples received were air dried and prepared for testing and analysis.

Material Characterization (Index properties)

Two separate samples of each soil were tested and tests were performed according to the standard ASTM procedures (ASTM D854-14, ASTM D6913-17, ASTM D7928-21e1, ASTM D4318-17, ASTM D698-12, ASTM D5918-13e1). The index properties and material classifications are given in Table 1.

Organosilane Selection

There are several products available and utilized in literature for imparting hydrophobicity to soils, namely Dimethyldichlorosilane (Ng and Lourenco, 2016), Wax (Bardet et al, 2011), Zycosoil (Trimethyloxysilyl) (Choi et al, 2017), Stearic acid (Leelamanie et al, 2008) and Tung Oil (Zhang et al, 2015) have been found effective in engineering water repellency in soils. While a number of products abound to impart hydrophobic properties to materials, three organosilane chemicals were selected for this study, considering their effectiveness (surface modification, reaction time, dosage), environmental safety (toxicity, solvent type, leaching properties), cost as well as other considerations (ease of handling, familiarity of use, fit to existing methods/machinery).

Table 1. Summary of Soil Index Properties and Classifications

	NC-BO	IA-PC	AK-FB
Specific Gravity, Gs (ASTM D854-14)	2.67	2.77	2.65
Liquid Limit, LL (ASTM D4318-17)	38.37	35.4	40.6
Plasticity Index, PI	NP*	12.1	NP*
Silt content (%) (75µm–2µm)	34.0	86.28	75.66
Clay content (%) (< 2µm)	4.88	12.08	8.86
Optimum Moisture Content (%) (ASTM D698-12)	15.56	16.8	20.9
Max. Dry Unit Weight (kN/m3) (ASTM D698-12)	15.92	16.4	14.8
USCS Classification	SM/SC	CL	ML
AASHTO Classification	A-5	A-6	A-5

NP* - Non-Plastic

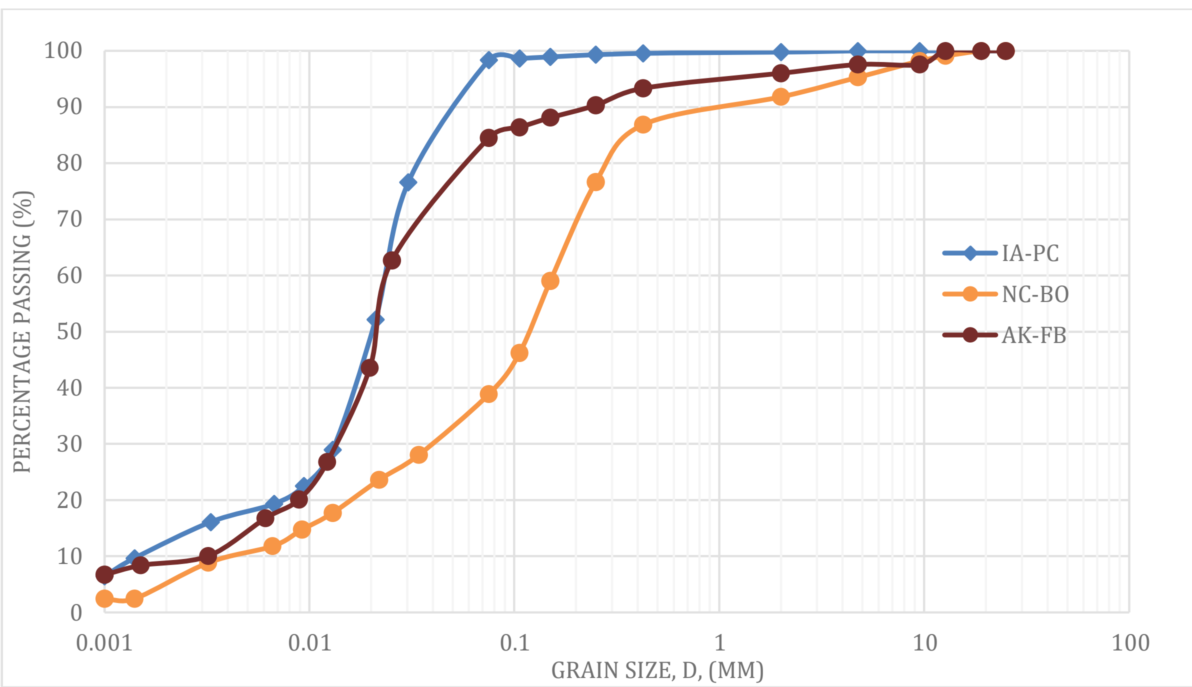

Figure 1: Grain Size Distribution of the Soil samples (ASTM D6913).

SIL-ACT® ATS-100 is a premium grade, clear, penetrating silane-waterproofing treatment. It complies with EPA and most state's VOC regulations and is adopted by a number of State Department of Transportation (DOTs). It has been used in areas such as bridge decks, highways, parking garages. (ATS-100 Product Datasheet; Ley et al, 2015). DOWSIL™ IE 6683 is a silane/siloxane emulsion blend, dilutable in water to formulate a less than 200 g/l VOC water repellent. It is particularly suited to porous construction materials such as bricks, stone, concrete blocks, mortar and grout. The ability to dilute and the varying-sized active ingredients ensures good penetration into porous substrates, resulting in particle coverage and durable water repellency (DOW Technical Sheet; Daniels et al. 2009; Keatts et al. 2018). Zydex Terrasil is a water-soluble and reactive soil modifier that permanently modifies the soil surface, making it hydrophobic. Terrasil is a non-leachable and safe chemistry, and works with all soil types. It has been utilized in the stabilization of soft clays and expansive soils (Terrasil Technical Sheet; Meeravali et al., 2020; Ewa et al., 2016; Zahoor & Jassal, 2020; Singh, 2017; Patel et al., 2015), with the net result of making soils water resistant and improving soil compaction (Oluyemi-Ayibiowu and Uduebor, 2019).

Chemical Treatment

Chemical treatment followed batch-type procedures as described in Roy et al. (1992) (Bachmann et al., 2000). Frost susceptible soils were oven-dried prior to treatment with the three OS products. The ratios selected were informed based on previous studies or literature with reduction in OS concentration to identify the resulting changes. Soils were treated with SIL-ACT ATS-100 (Advanced Chemical Technologies) at a ratio of 1:1, 1:6, 1:12, 1:24, 1:50 and 1:100 (OS to soil, batched by weight) in a 250ml HDPE bottle and set up on a tumbler to react for 24 hours. For DOWSIL IE6683 (Dow Chemicals), 1:10, 1:20, 1:40, 1:80, 1:160 (OS to soil, batched by weight) and for Terrasil (Zydex Industries) mix ratios of 1:10, 1:20, 1:40, 1:80, 1:160, 1:500 and 1:1000 (OS to soil, batched by weight) were selected and the OS mixed with Deionized (DI) water to achieve a Liquid/Solid Ratio of 1:1 to ensure sufficient saturation of the soil samples. Therefore, for a 50g of oven dried FSS sample, the OS quantity was diluted in DI water to make up 50g. The diluted OS as well as the soil sample were mixed vigorously by hand for one minute in a 250ml HDPE bottle and set up on a laboratory tumbler to react for 24 hours.

The resulting mixture was then separated into two cans, one air dried and the other dried in an oven at 60°C. Oven-dried samples were allowed to dry for 24 hours and then allowed to cool for 24 hours to prevent a pseudo-enhancement of the hydrophobicity of the soil due to heat treatment (Roy et al, 1992). Where there were lumps or clusters of bonded soil, a mortar and rubber-tipped pestle were used to break the OS treated soil before testing.

Contact Angle (CA) Test

The contact angle test is a widely accepted method for assessing the degree of water repellency of solid surfaces (Adamson, 1990). Bachmann et al. (2000) developed the sessile drop contact angle method (SDM) to determine contact angles for soils.

A double-sided adhesive tape was attached to a glass slide and oven dried samples (60ºC) were sprinkled on the other coated side and compressed for 10s. 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 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) and drops of deionized water were placed on the surface of the specimen, gradually advancing the drop size with continual horizontal image capturing and measurements were taken within 10s for each drop size. Contact angle measurements less than 90^o are considered wettable/hydrophilic, while angle measured between 90^o and 150^o are considered hydrophobic. Angles above 150^o are taken to be super hydrophobic.

Water Drop Penetration Test (WDPT)

The water drop penetration test (Feyyisa et al., 2017) was utilized to assess the water repellency of bulk sample mass. About 20g samples in sample cans were used for the WDPT measurements. Three drops of deionized water (50 ± 1 μL volume) were placed on the soil surface with a burette. The tests were conducted at night and under constant temperature (~25^{o}C) without draft to minimize evaporation during the experiment. A video recording (with timecode stamp) of the complete penetration of the water drop was utilized to determine the time taken. Instantaneous penetration was taken to be 0.2s and all measurements were terminated after 1 hour (3600s) and all WDPTs exceeding 3600s were assigned as extremely water repellent. Table 2 presents repellency categories (King 1981; Liu et al. 2012; Bisdom et al. 1993; Chenu et al. 2000) based on corresponding WDPTs.

Table 2: Water Repellent Categories for Water Drop Penetration Test

Water Drop Penetration, s	Repellency category
≤ 1 s	Non-repellent/Completely wettable
1–60 s	Slightly repellent
60–600 s	Strongly repellent
600–3600 s	Severely repellent
≥ 3600 s	Extremely repellent

RESULTS AND DISCUSSION

Figure 2 below shows the contact angle measurements for both air-dried and oven-dried treated samples. It is noticeable that the contact angles from the air-dried samples are close to those of the oven-dried samples. While the air-dried samples results are more typical of field treatments, the oven-dried samples give the maximum resulting hydrophobicity from the treatment of the soils.

Figures 3a-c shows the soil-water contact angles of the modified water repellent FSS. All OS chemicals were able to impart sufficient hydrophobicity to all the soil samples well above the 90^o and close to super hydrophobicity (150^o). Similar studies carried out on hydrophobized soils observed a similar trend (González-Peñaloza et al, 2013; Saulick et al. 2021). The treatment using the ATS-100 and Terrasil have better effectiveness in imparting water repellency properties to the soils with values significantly greater than 90^o.

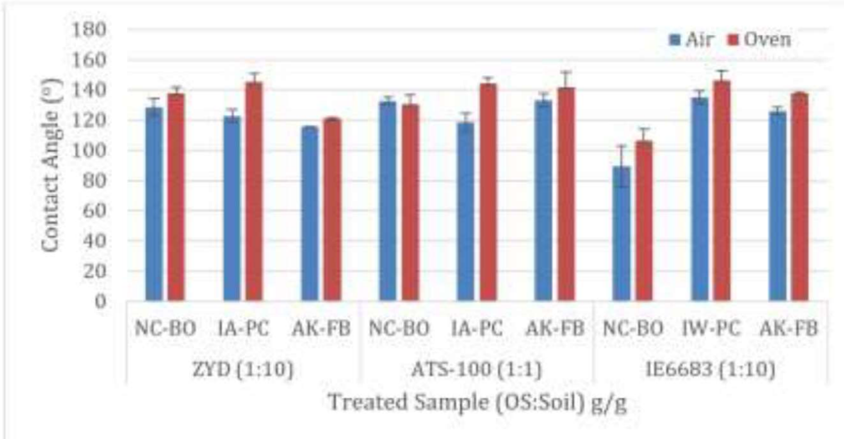

Figure 2: Measured Soil-Water Contact Angles for Air-Dried and Oven-Dried Treated Samples.

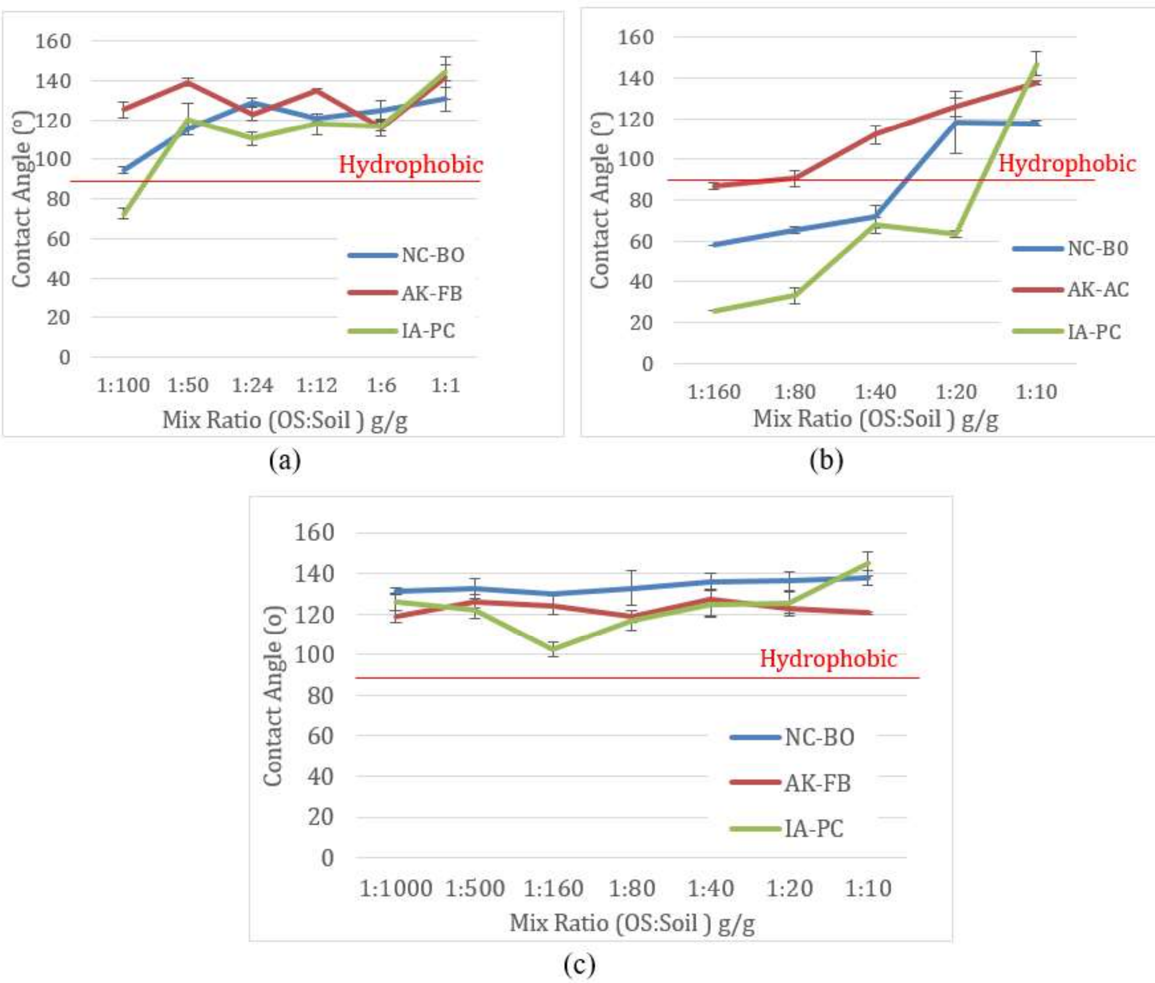

Figure 3: Measured Soil-Water Contact Angles with Different OS Treatment at Different Concentrations (a) ATS-100 (b) IE6683 (c) Terrasil.

Based on analysis of an idealized heterogeneous surface, Johnson and Dettre (1964) reported that the contact angle is almost constant when the coverage of non-wettable regions exceeds 40% of the total surface. From the almost unchanging values of the contact angle measurements, it can be implied that the soil particles of the NC-BO and AK-FS soil were sufficiently covered with OS (1:50 for ATS-100 and 1:1000 for Terrasil), while that of IA-PC kept increasing implying insufficient coverage. According to Choi et al. (2016), a constant contact angle does not necessarily imply a maximum amount of grafted OS and the degree of water repellency could further increase with increase in OS concentration. It is important to note that different soils will react differently to OS chemicals, hence it is important to always establish a baseline result from experiments before utilizing any product. In practical applications, a specified contact angle and corresponding water entry head, should be the aim in determining appropriate mix-ratios.

The effect of increasing OS concentration on WDPT is presented in Figures 4a-c. The WDPTs of specimens treated with ATS-100 and Terrasil were ≥3600s (extremely repellent), with water droplets still on the surface after 2 hours. While for treatment with IE 6683, the WDPTs of treated samples became extremely repellent at a mix ratio of 1:40 for both NC-BO and AK-FB and at 1:10 for IA-PC. It is important to note that AK-FB has some natural water-repellent properties.

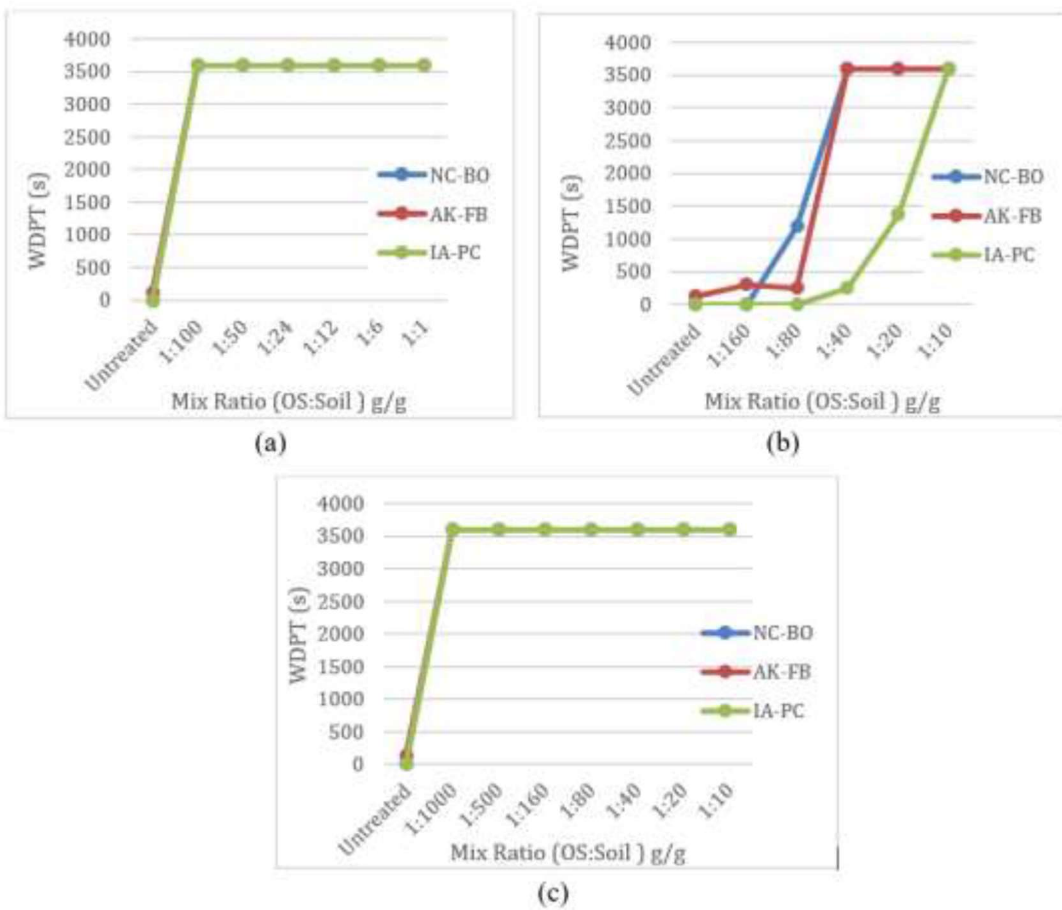

Figure 4: Measured Soil-Water Contact Angles with Different OS Treatment at Different Concentrations (a) ATS-100 (b) IE6683 (c) Terrasil.

CONCLUSIONS

This study investigated the treatment of selected frost susceptible soils using commercially available OS chemicals at varying concentration dosages to impart water repellent properties. The degree of hydrophobicity imparted, and performance were determined using contact angle measurements as well as waterdrop penetration testing. Treatment using ATS-100 and Terrasil were found to be effective even at small concentrations (1:100 and 1:1000 respectively) with values ranging from 73° to 144° for ATS-100 and 103° to 145° for Terrasil. Effective values for DOWSIL IE6683 were obtained at a ratio of 1:20 for NC-BO and AK-FB while a ratio of 1:10 was established for IA-PC. The results obtained correlated with WDPT results.

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