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Compaction Characteristics of Organo-silane Treated Soils

TECHNICAL NOTE

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ABSTRACT

The extent to which hydrophobic soils can be used in geotechnical engineering practice depends upon a commensurately increased understanding of how fundamental relationships apply in hydrophobic systems, such as moisture-density. This research is to determine the compaction characteristics of organo-silane (OS) treated soils using conventional geotechnical laboratory equipment. The intrinsic difficulty in lubricating hydrophobic soils, which would allow for rearrangement of particles and subsequent compaction, arises from their inherent low surface energy. This work describes a method for compacting OS treated soils that leverages the necessary conditions such as reaction time and drying conditions for achieving hydrophobicity. Procedural steps for compacting OS treated soils are detailed by making use of a water-soluble hydrophobizing agent added to a fine-grained soil. Based on the critical dosage ratio of 1:100 (hydrophobizing agent: soil) identified, a molding water content is defined constituting of a fraction of the hydrophobizing agent. Soil water content and dry density curves are developed using the standard Proctor and Harvard miniature to contrast the resulting effect of OS. Compared to the untreated soil, a decrease in optimum water content was observed with the OS treated soil regardless of compaction technique used. For the standard Proctor test, a decrease in optimum water content from 12.0 to 9.2 % was observed, whereas compaction with the Harvard miniature showed a marginal decrease from 9.3 to 9.0 %. With the untreated soil, a relatively larger maximum dry density (2.17 g/cm³) was obtained with the standard Proctor compared to the Harvard miniature (2.05 g/cm³). The protocol defined to compact OS treated soils has shown to induce hydrophobicity spatially within the sample depth. These results suggest that engineered water repellency can be implemented in so far as treatment and compaction are largely synchronous and prior to reaction and hydrophobization.

Keywords

contact angle, soil density, water repellency, fine-grained soils, engineering properties

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Introduction

Water is the source of virtually every challenge in geotechnical practice. Uncontrolled water content corresponds to reduced strength, increased swell, and greater erosion, among other undesired effects. In many geotechnical systems, a specified degree of compaction is used to ensure a given level of performance. The process of soil compaction is dependent on mineralogy, particle size and shape, water content, compaction energy as well as properties of the fine grains such as the liquid limit (Van Der Watt 1969; Reddy and Jagadish 1993; Sivrikaya, Togrol, and Kayadelen 2008; Kuriakose et al. 2017; Karakan and Demir 2018; Cabalar, Khalaf, and Isik 2020; Karakan, Shimobe, and Sezer 2020; Karakan and Demir 2020; Karakan 2022a, 2022b). Two geotechnical parameters of main interest in the process are the maximum dry density and optimum water content. Compaction carried out at maximum dry density and optimum water content ensures maximum densification with the minimum amount of water and theoretically corresponds to the utmost air exclusion from the soils (Ren et al. 2015).

Laboratory compaction of soils provides valuable insights into the soil behaviour in a controlled environment, thus facilitating the standardization of protocols. The Proctor test is by far the most commonly carried out test for compaction of soils. Several variations of the test have been documented in the literature, for instance, a modified version of the test (modified Proctor) making use of a relatively larger mold, a heavier rammer, and an increased number of blows and layers has been developed in the 1940s. Other changes to the original procedure include reducing the number of blows to align compactive effort carried out in the laboratory with field work (Daniel and Benson 1990). A comparatively quicker compaction method requiring less soil for compaction is the Harvard miniature test. Although compaction carried out with the Harvard miniature are able to more closely mimic field tests, their use has been very often restricted to supplementing Proctor tests (Loshelder, Chanis, and Coffman 2023). Possible reasons include the sensitivity of the resulting maximum dry density to the number of layers, tamps, and energy applied (Loshelder, Chanis, and Coffman 2023). Field and laboratory methods for compaction are predicated on the expectation that soils are hydrophilic, i.e., wettable. Such wettability reduces interparticle friction and enables particles to more easily rearrange into a denser configuration in response to the application of a force (kneading, rolling, or impact). In the case of hydrophobic or water repellent soils, an alternative approach is required to achieve density and associated criteria.

The concept of engineered water repellency as a response to limiting uncontrolled water content indicates that hydrophobic soils are able to withstand detrimental changes in geotechnical properties, e.g., a reduction in expansion of swelling clays (Hernandez et al. 2005), capillary rise (Orozco and Caicedo 2017), frost heave (Mahedi et al. 2020), and changes in electrical conductivity (Dong and Pamucku, 2015) have been documented. To render soil hydrophobic, several classes of compounds have been explored in the literature, e.g., fatty acids (Subedi et al. 2012), waxes (Bardet, Jesmani, and Jabbari 2011), oils (Lin et al. 2021), and pure silanes (Movasat and Tomac 2021). The use of pure silanes such as dimethyldichlorosilane in soil samples, characterized by a single-step process, offers a convenient means to impart hydrophobicity in addition to carrying a low environmental threat to microbiota (Lin et al. 2022). However, this attribute gives rise to a challenge for compaction of such soils because the process of compaction relies on adding water for lubrication and particle rearrangement. As such, hydrophobic soils by definition have an intrinsic resistance to absorption and uniform distribution of water during the compaction process. The nature of this resistance can be understood from the historical use of liquid mercury for determining pore volume and its distribution via mercury intrusion porosimetry (e.g., ASTM D4404-18, *Standard Test Method for Determination of Pore Volume and Pore Volume Distribution of Soil and Rock by Mercury Intrusion Porosimetry*) where mercury resists pore entry, which enables it to be used in pore diameter measurements. The resistance is a function of the contact angle that develops between liquid mercury and particle surfaces, which exceeds 90°. The same type of resistance and contact angle relationship develops between liquid water with a hydrophobic surface. In either case, the liquid cannot be homogeneously distributed across particle surfaces, resulting in heterogeneous compaction with wide localized variation in density. Possible techniques to enable compaction of hydrophobic soils may include the use of more

energy-intensive equipment or repetitive water addition and soil compaction, or both. However, such potential solutions can have several drawbacks when used in an engineering context; for instance, use of more energy intensive equipment may be cost-prohibitive and repetitive compaction may lead to waterlogged soil. Therefore, a technique that enables samples to be molded at a prescribed water content (allowing the maximum dry density and optimum water content to be identified) while overcoming limitations related to workability is needed.

In this work, a water-soluble hydrophobizing agent is used for compaction of soils. The aim of this article is to provide a guideline for obtaining compacted hydrophobic soils and overcoming challenges associated with workability using conventional laboratory compaction equipment. The work presented is the first to detail procedural steps to obtain compacted hydrophobic soils using the standard Proctor and Harvard miniature.

Materials

SOILS

Soil from Hanover, New Hampshire, was used in this study. **Figure 1** depicts the particle size distribution of the soil, and **Table 1** shows its classifications and characteristics such as specific gravity as per ASTM D854-14, *Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer* (Withdrawn 2023). Samples were

FIG. 1 Particle size distribution of soil.

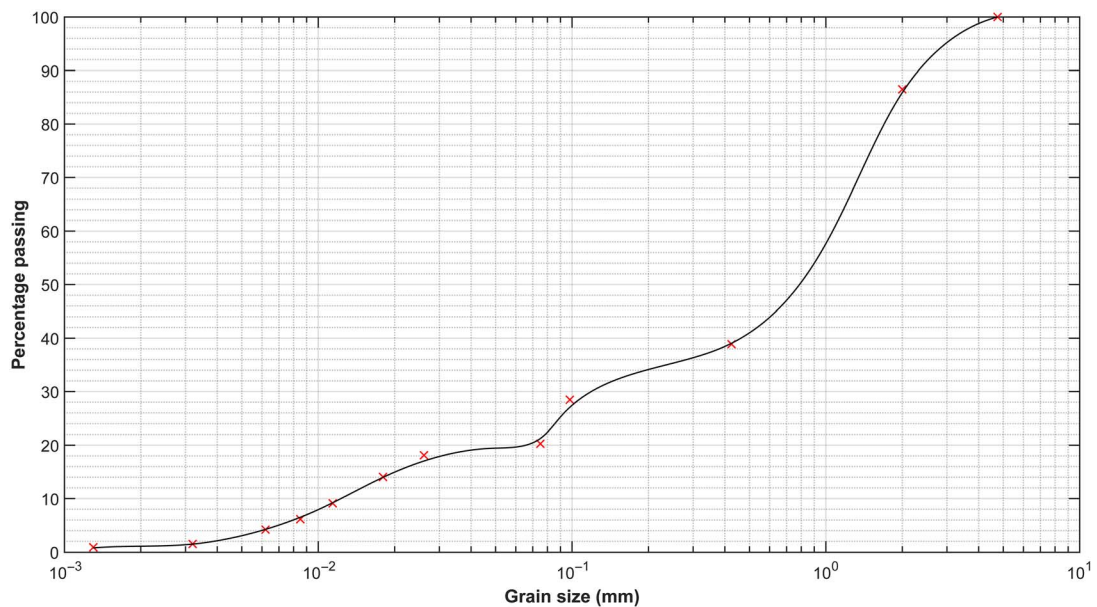


TABLE 1

Characterization of Hanover, New Hampshire soil

Unified soil classification system	SM
AASHTO soil classification system	A-5
Specific gravity	2.68
Percentage passing no. 200 sieve (75 μ m)	20.3
Liquid limit	22.8
Plastic limit	NP

prepared by first oven-drying at a temperature of 105°C, after which they were manually separated for a dry and wet sieving process. Soil retained on the no. 4 sieve (4.75 mm) was discarded (**fig. 2A**) and the resulting passing soil (**fig. 2B**) was homogenized and batched into 300-g samples for subsequent testing.

HYDROPHOBIZING AGENT AND HYDROPHOBIC CHARACTERIZATION

There exists a variety of hydrophobizing agents having silanes as their primary constituents that are commercialized by a growing number of companies, targeting numerous applications such as textiles, automotive, and construction. For this research, a product marketed as TerraSil was chosen; it is a water-soluble organo-silane (OS) in liquid form from Zydex Industries. The OS product contains approximately 65 % of alkoxy-alkylsilyl compounds that form outward-oriented functional groups that are responsible for inducing hydrophobicity. The solvent within the OS product used to dispense the hydrophobic compounds is predominantly benzyl alcohol (25 %). **Table 2** and **figure 2C** show the physical and chemical properties of the OS product. To impart

FIG. 2 (A) Soils discarded during sieving, (B) soils retained and used during tests, (C) OS product, and (D) soils compacted using the Harvard miniature test at 8 % and 18 % water contents (sample heights are approximately 7.2 cm).

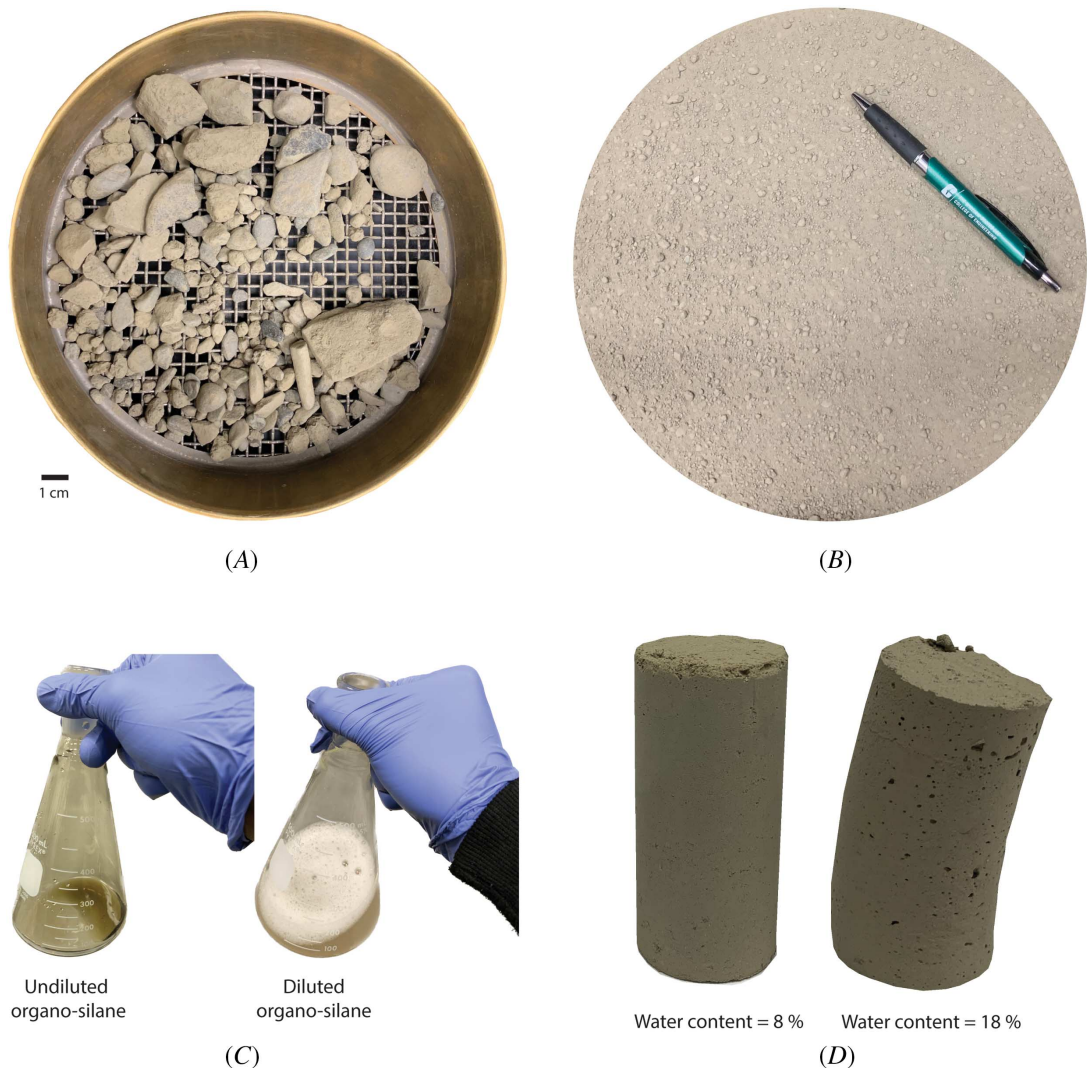


TABLE 2

Physical and chemical properties of the OS product

Color	Pale yellow
Odor	Aromatic
Melting point	6°C
Boiling point	200°C
Viscosity	0.1–0.5 Pa·s (at 30°C)
Density	1.05 g/cm ³

hydrophobic properties to substrates, the OS product needs to be first diluted in water and unlike pure silanes such as dimethyldichlorosilane, the resulting diluted OS product-soil mixture needs to be either oven or air-dried before displaying hydrophobicity as shown in Brooks et al. (2022). In this study, samples of the OS product-soil mixture were oven-dried at 105°C.

A goniometer (Ramé-Hart Instruments, 260-1, standard goniometer, #150512) was used to measure the contact angles of the materials using the sessile drop method, a method widely used to assess the hydrophobicity of granular media. Sample preparation of each soil was carried out according to the technique proposed by Bachmann, Ellies, and Hartge (2000) by fixing a monolayer of soils on a microscope slide with double-sided tape attached to it, and 10- μ l drops were dispensed on the samples using the FlowTrac II (Geocomp Products). The semi-automated technique developed by Saulick, Lourenço, and Baudet (2017) was applied to evaluate the contact angles using ImageJ, an open source image processing software. The mean value of ten measurements and the corresponding standard deviation on each sample were adopted as the measured data.

Procedural Steps for Compaction of OS Treated Soils

To obtain the dry density-water content relationship of the soils, a range of water contents spanning the dry and wet sides of the estimated optimum water content was investigated with both the standard Proctor and the Harvard miniature tests. Decreasing water content from 20 % was used to visually and tactilely demarcate the upper bound water content to be tested. For example, a water content of 18 % was deemed too high for compaction tests and had a spongy texture as opposed to a water content of 8 % (fig. 2D). In this study, the water content (ranging from 5 to 16 %) was adjusted by adding a calculated amount of water and thoroughly mixing the soil until the targeted water content was uniformly distributed.

CHARACTERIZATION OF HYDROPHOBICITY

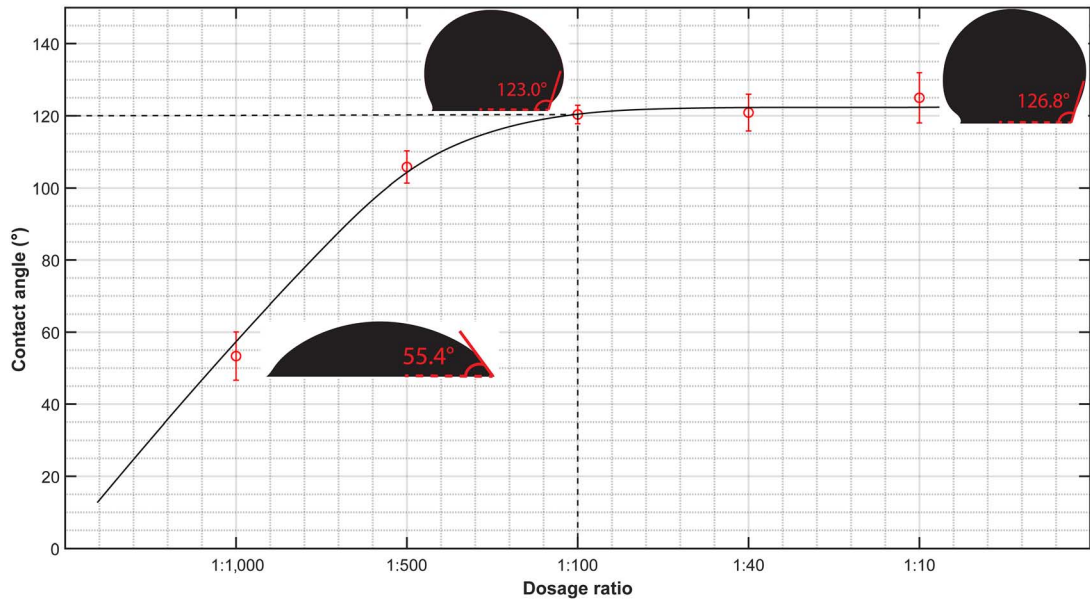
To render the soils hydrophobic, both the OS product and water need to be added to the soil. For clarity in the subsequent texts, the terms dilution ratio, relating the mass of OS product to water and dosage ratio, relating the mass of OS product and water to soil are used.

Based on the previous studies carried out by Uduebor et al. (2022) and Brooks et al. (2022), the following dosage ratios were selected: 1:10, 1:40, 1:100, 1:500, and 1:1,000 for the characterization of hydrophobicity using the OS product. Using a dosage ratio of 1:100 as an example, the steps to diluting the OS product are as follows:

1. Assume a solution (OS product plus water) to soil ratio of 1:1
2. Mass of solution and mass of OS product required are 100 g and 1 g, respectively
3. Dilution ratio of OS product to water is 1:99

Figure 3 illustrates the relationship between the dosage ratio of diluted OS product and contact angle of the soil. Increased addition of the OS product from a dosage ratio of 1:1,000 (lesser amount of OS product) up to 1:10 enhances the hydrophobicity of the soil up to a limit (see plateaued region), beyond which, further addition of OS product does not significantly change contact angle. This dosage ratio, referred to as critical dosage is

FIG. 3 Relationship between dosage ratio and contact angle for soil. Error bars are standard deviations of the mean (10 measurements). Inset photographs illustrate representative 10- μ l drops on samples at dosage ratios of 1:1,000, 1:100, and 1:10. Dashed lines represent dosage ratio beyond which no significant change in contact angle is observed.



dependent on the inherent characteristics of soils such as mineralogy, particle size, and the dosage ratios investigated. In this work, a dosage ratio of 1:100 resulting in a contact angle of 120° was selected as the critical dosage.

COMPACTION OF OS TREATED SOILS

For the compaction of OS treated soils, the resulting dilution ratio is not only a function of dosage ratio but also dependent on the water content at which compaction is carried out. The following details the steps to determine the dilution ratio for compacting 120 g of soil at a water content of 15 %:

1. Determine the critical dosage ratio, e.g., 1:100
2. Mass of solution and mass of OS product required are 18 g (15 % of 120) and 1.2 g, respectively
3. Dilution ratio of OS product to water is 1: $\frac{18-1.2}{1.2} = 1:14$

Standard Proctor

The steps undertaken for compacting the soils using the standard Proctor equipment as per ASTM D698-12, *Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort* (12, 400 ft-lbf/ft³ (600 kN-m/m³)), are briefly described as follows:

1. After weighing the empty mold (diameter 10.2 cm and height 11.6 cm), the apparatus was set up on the base plate and with the collar.
2. A total of three layers (thickness = 3.9 cm each) was used in the mold with each layer scarified before the addition of the next layer.
3. A 2.5-kg rammer was dropped from a distance of 30 cm for a total of 25 blows in each layer. The first four tamps were applied in separate quadrants of the mold and the fifth tamp was applied in the center.
4. After compaction, the extension was removed and excess soil was trimmed away. The mold was then re-weighed to obtain the mass of soil.

5. A specimen ejector was then used to extrude the compacted soil, from which 30 g of soil were sampled at three locations to obtain the water content after oven drying at 105°C for 24 h.

Harvard Miniature

The Harvard miniature testing apparatus ([fig. 4](#)) consists of a 17-kg tamper that is equipped with a spring, characterized by a spring constant of 1.69 N/mm (Humboldt Mfg. Co.). By adjusting the potential energy of the tamper via the displacement of the spring, the test can be calibrated to deliver an equivalent energy level as imparted by the standard Proctor test. The Harvard miniature apparatus was set up to determine the compaction properties of the soils following the method proposed by Wilson ([1970](#)). This is briefly outlined as follows:

1. After weighing the empty mold (diameter 3.3 cm and height 7.2 cm), the apparatus was set up on the base plate and with the collar.
2. A total of five soil layers was used in the mold. Each soil layer (of thickness approximately equal to 1.5 cm) was levelled prior to compaction and scarified before the addition of the next layer.
3. The tamper was used for compacting each layer in the mold, with a total of 25 tamps done in each layer. The first four tamps were applied in separate quadrants of the mold and the fifth tamp was applied in the center.
4. After compaction, the collar was removed and excess soil was trimmed away. The mass of soil was obtained by subtracting the mass of the empty mold from the mass of mold and compacted soil.

FIG. 4 (A) Harvard miniature compaction equipment, (B) tamper assembly, (C) mold assembly, and (D) specimen ejector.



5. The specimen ejector was then used to extrude the sample and the extruded sample was oven-dried at 105°C for 24 h before determining the water content gravimetrically.

Based on the analysis of dosage ratios presented in [figure 3](#), compaction was carried out at the critical dosage ratio of 1:100, characterized by negligible fluctuation in contact angle. The solution of OS product and water was used as the molding water content for the compaction process. To ensure proper conditioning, after the addition of diluted OS product to the soils, they were sealed in plastic bags for 24 h prior to testing.

DRY DENSITY AND WATER CONTENT RELATIONSHIP

The dry density and water content relationships carried out with the standard Proctor test for the untreated and OS treated soils are shown in [figure 5A](#). The magnitudes of dry density determined for the untreated soil lay above the OS treated soil with a difference of at most 0.215 g/cm³. A decrease in optimum water content (from 12.0 to 9.2 %) was recorded when comparing untreated to OS treated soils. In comparison, with the Harvard miniature, no distinct change in the compaction characteristics were observed between the untreated and OS treated soils ([fig. 5B](#)): a marginal decrease in optimum water content from 9.3 % (untreated soil) to 9.0 % (OS treated soil) was observed with differences in maximum dry densities of less than 0.2 %. These results are consistent with previous reports such as Daniels et al. (2009) and Malisher et al. (2023) who indicated that the use of OS products for inducing hydrophobicity can be expected to lower the optimum water content.

On the other hand, the results of maximum dry density suggest that these values depend on the compaction methodology and soil properties. With the untreated soil, a maximum dry density of 2.17 g/cm³ was obtained with the standard Proctor compared to 2.05 g/cm³ with the Harvard miniature. This difference is attributed partly to the relatively smaller area (1.28 cm²) of the tamper rod of the Harvard miniature, which penetrates underlying soil layers leading to lower dry densities. This disparity could potentially also be linked to the soil properties investigated, e.g., Loshelder, Chanis, and Coffman (2023) showed that when comparing soils with different liquid limits, such as one with a liquid limit of 27, the maximum dry densities were similar, whereas a soil with a liquid limit of 37 showed a relatively higher maximum dry density using the standard Proctor method.

[Figure 6A](#) and [6B](#) illustrate the soils compacted with the standard Proctor at a water content of 9.39 % before and after oven drying at 105°C, respectively. Water drops dispensed on the compacted hydrophobic soils before oven-drying infiltrated instantaneously, whereas samples investigated after oven-drying showed drops beading on the surface of the compacted soil, demonstrating the feasibility of the protocol in inducing hydrophobicity in compacted soils. Similar observations were made with samples compacted with the Harvard miniature. [Figure 6C](#) illustrates that the proposed compaction methodology is able to induce hydrophobicity spatially within the soil depth.

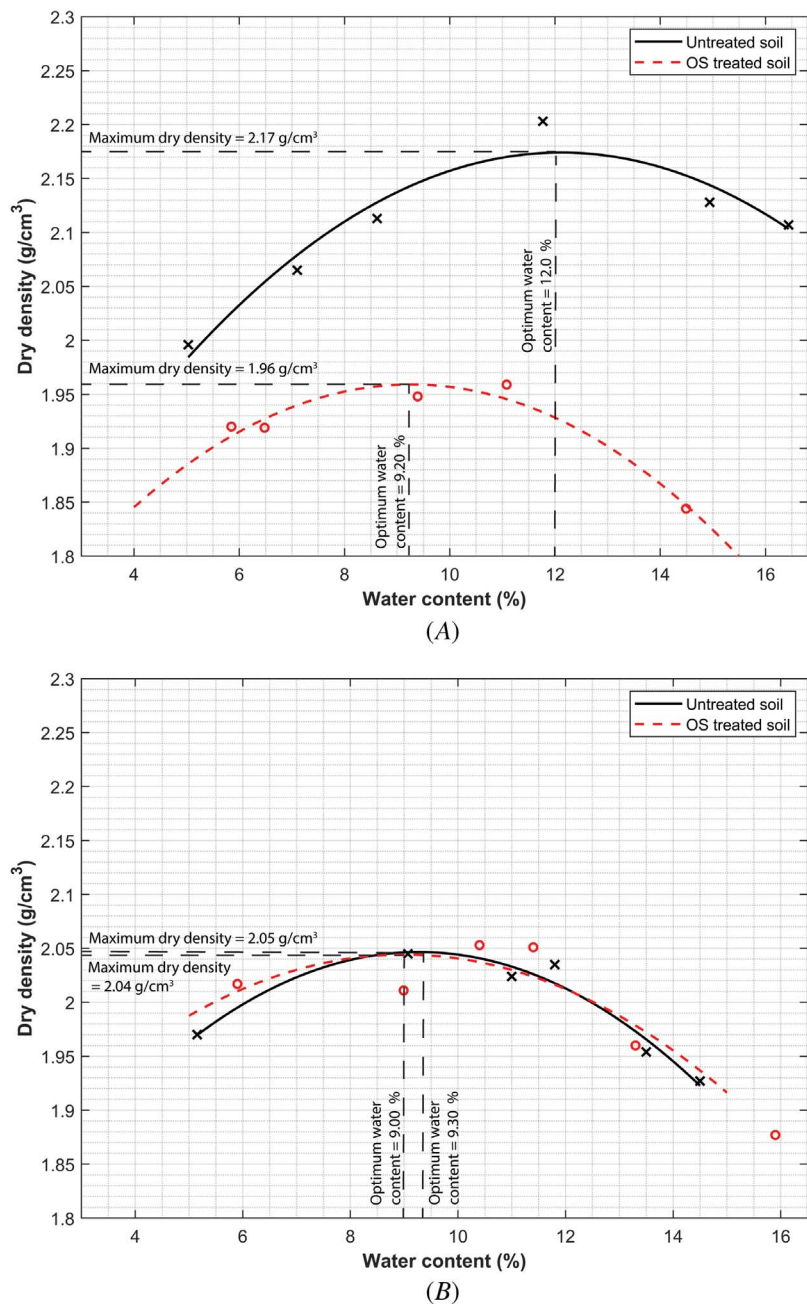
Practical Significance

ENGINEERING APPLICATIONS OF HYDROPHOBIC SOILS

The implementation of hydrophobic soils as capillary barriers ([Subedi et al. 2012](#)), slope covers ([Zheng et al. 2017](#)), part of a pavement system ([Mahedi et al. 2020](#)), and subsurface layers of sport tracks ([Bardet, Jesmani, and Jabbari 2011](#)) necessitate a level of compaction to achieve targeted geotechnical properties such as shear strength and permeability. Other still unexplored applications such as foundation design, utility protection, hydrocarbon adsorption/filtering, and coastal erosion mitigation will also need dry density-water content relationship of hydrophobic soils to optimize their use for their respective applications. For instance, in foundation design, to mitigate the influence of high fluctuating groundwater levels and the presence of saline water (which can lead to deformation and erosion, and deteriorate soil-structure interaction), a relatively high soil density is necessary. The laboratory tests presented in this article provide a baseline for using hydrophobic soils in construction to ensure that a targeted level of compaction or water content, or both, is obtained.

FIG. 5

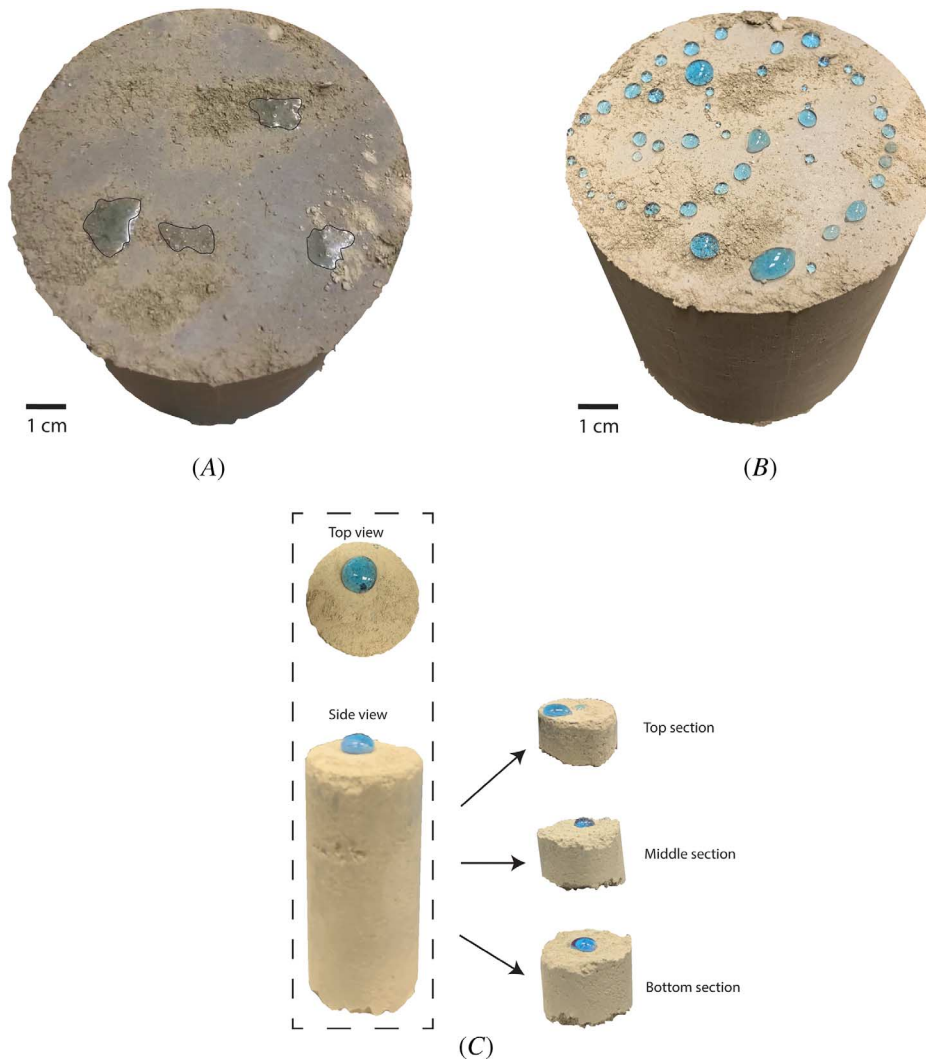
Comparison of
(A) standard Proctor and
(B) Harvard compaction
tests for untreated and
OS treated soils.



DRYING CONDITIONS

The use of the compaction method detailed in this study requires accounting for reaction time between the OS product and the soil to generate hydrophobic coatings. This is accompanied by simultaneous drying of the treated soil to display hydrophobicity as illustrated in [figure 6](#). In situ application of such a protocol will require loss of moisture through processes such as evaporation and percolation. Factors that will govern drying under field

FIG. 6 Soil compacted with (A) standard Proctor before oven-drying. The water drops infiltrate immediately once dispensed on the surface. (B) standard Proctor after oven drying. The water drops bead up on the surface of the soil. (C) Harvard miniature depicting the spatial distribution of hydrophobicity. The sample height is approximately 7.2 cm.



conditions include climatic conditions where hydrophobic soils are being utilized (e.g., dry, tropical), the depth at which the hydrophobic soils are to be implemented and soil texture.

OTHER ENGINEERING CHARACTERISTICS

Achieving the desired level of compaction is one of the necessary characteristics for design considerations with hydrophobic soils. Other equally important characteristics that are intricately linked to the compaction characteristics include water entry pressure and retention behavior of hydrophobic soils. The former is the critical pressure at which water displaces air and the latter is a characteristic that differs significantly from untreated soils, having a lower degree of saturation for a given soil potential (Czachor, Doerr, and Lichner 2010). Both the water entry

pressure and retention behavior of hydrophobic granular media are functions of the pore characteristics, which depend on their density (Bauters et al. 2000; Lee et al. 2015; Keatts et al. 2018).

Conclusions

This work offers qualitative and quantitative guidance for developing moisture-density relationships relevant to hydrophobic soils. In particular, we recommend that laboratory or field-based compaction be carried out prior to hydrophobicity being exhibited by the soil, analogous to lime or cement treatment in which cementitious reactions cure post-compaction. Otherwise, and by definition, the formation of low surface energy coatings on the surfaces of soil particles will influence their homogeneous rearrangement to increase density. The results reported in this study are consistent with previous literature documenting the effect of OS treatment on the compaction characteristics of granular materials where a lower water content is to be expected for the maximum dry density when hydrophobicity is induced. However, comparison of compaction characteristics with the standard Proctor and Harvard miniature suggests that the maximum dry density is a function of compaction methodology (impact or kneading) and soil properties.

Despite the proposed use of hydrophobic soils in several applications, design protocols to achieve targeted fundamental engineering properties as documented in this work is still lacking. In general, the acquisition of compaction parameters is a time-consuming procedure and to be able to transition to using empirical models (e.g., correlations between compaction parameters and index tests) for preliminary design stages of projects, a database of compaction parameters for hydrophobic soils is needed. The design protocol presented establishes a foundation to achieve a seamless testing methodology with hydrophobic soils.

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