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Online Publication Date: 20 May 2026

URL: <http://www.jresm.org/archive/resm2026-1640ma0426rs.html>

DOI: <http://dx.doi.org/10.17515/resm2026-1640ma0426rs>

Journal Abbreviation: *Res. Eng. Struct. Mater.*

To cite this article

Jassam M G, Abd A H, Al-Sumaiday H G. Collapsibility and strength of gypseous soil waterproofed by silane coupling agent. *Res. Eng. Struct. Mater.*, 2026; 12(3): 1943-1954.

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Research Article

Collapsibility and strength of gypseous soil waterproofed by silane coupling agent

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Article Info

Abstract

Article History:

Received 26 Apr 2026

Accepted 19 May 2026

Keywords:

Gypseous soil;
Water repellent;
Collapsibility;
Waterproofed soil;
Silane coupling;
Soil additives

Water has long been a major source of instability and deterioration in the strength of gypseous soil. This study examines the effect of adding a water-repellent agent on the collapsibility and shear strength of gypseous soil. This silane agent (also known as KH-570) is a low-viscosity organic silane solution that imparts water-repellent properties to surfaces and materials. Samples of disturbed soil containing 45% gypsum were collected from Salah Al-Din Governorate in Iraq at a depth of 1.5 meters below the natural ground surface. The additive concentration was selected by spraying and mixing with soil samples at 2%, 4%, and 6% by weight of the soil used. Consolidation and direct shear tests are conducted for both treated and untreated soil samples. Results show significant improvement in collapse potential values for treated soil in comparison with untreated ones. It was found that the optimal weight ratio for the additive is 2%, and this value is used when calculating shear resistance coefficients. In the case of semi-dry soil, the cohesion strength and the angle of internal friction increased by 89% and 9.3%, respectively, and for the soaked case were 119% and 250% respectively compared to the untreated soaked sample. Although KH-570 may have a relatively higher unit cost compared with traditional stabilizers such as lime or cement, the low required dosage (2%) and the significant improvement in waterproofing as well as reduce the long-term maintenance costs make it a cost-effective stabilization technique for gypseous soils.

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1. Introduction

Gypseous soil, characterized by a considerable amount of calcium sulfate dehydrate. It is widespread in arid and semi-arid regions around the world. Its behavior depends on its gypsum content, and it loses much of its strength when saturated [1]. This soil type presents significant engineering challenges due to its susceptibility to collapse and loss of strength upon hydration. The wetting of gypseous soil can lead to gypsum decay which increase voids volume and permeability of soil [2]. Therefore, many studies have been conducted for increasing its stability by different treatments. This literature review discusses the different stabilization methods and stabilizers that were used to overcome the negative impacts of gypseous soil properties.

The use of lime as a stabilizer for gypseous soil is one of the earliest stabilizers used because of its availability and cheapness. This potential of lime to reduce metastability of gypseous soils was indicated by [3]. Al-Sheakayree studied the shear strength behavior of lime treated gypseous soil and concluded that the optimum dose of lime is 1.5% after 7 days [4]. Similarly, Ibrahim et al. examined the use of 5% hydrated lime, among other stabilizers, and concluded that lime effectively reduces soil collapsibility, making it a suitable choice for soil improvement [5].

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DOI: <http://dx.doi.org/10.17515/resm2026-1640ma0426rs>

Res. Eng. Struct. Mat. Vol. 12 Iss. 3 (2026) 1943-1954

The incorporation of lime with other stabilizing agents has also been a focus of recent research. Moayyeri et al. investigated the effect of combining lime and silica fume in gypsum soil. Their study involved mixing the soil with 1–3% lime and 1–7% silica fume. They observed that increasing the curing time enhanced compressive strength, with soils treated with 2–3% lime and varying proportions of silica fume exhibiting greater strength and durability after curing. This suggests that the combination of lime and silica fume may be a promising stabilization method [6]. Majeed also found that unconfined compressive strength and maximum dry density improved with an increase in the mixture of sugarcane ash and lime at effective ratios of 5% lime and 7% sugarcane ash [7]. Jha and Sivapulliah concluded that fly ash can be effectively used to control the negative effects of gypsum in lime-treated soil, as the use of 20% fly ash improves the volume change behavior of lime-treated soil in the presence of different amounts of gypsum, and this is considered the optimal ratio [8]. In addition, Mohammed and Elsageer show that the compaction properties and density of sandy soil is improved by adding lime and steel slag [9]. Adekanle et al. found that the soil's unconfined compressive strength after a 7-day treatment period increased with the addition of a mixture of lime and wheat straw, the highest value of CBR was found for 6% lime and 2% wheat husk [10]. Muhawiss and Hassoun concluded that adding cement to gypseous soil drop the collapse potential by 65% and increase cohesion by 50% [11].

In addition to lime stabilization, chemical grouting has been explored as a technique to enhance soil properties. Fattah et al. studied the effects of acrylate liquid grouting on undisturbed gypseous soil samples. Their results showed a reduction in soil compressibility by about 60% due to the isolating properties of acrylate. Additionally, soil cohesion increased, though the internal friction angle decreased. This study underscores the effectiveness of chemical grouting in modifying soil behavior, particularly in reducing compressibility [12]. Taha et al. show that the increase in unconfined compressive strength for enhanced gypseous soil reaches 33% with adding 6% of liquid asphalt and the strength decrease after this percent [13]. AL-Neami demonstrated that adding 4% of clinker to gypseous soil with 40% gypsum content was optimal for enhancing collapsibility and shear strength [14]. Abed examined the effect of a mixture of emulsified asphalt and dense sand on the engineering properties of gypseous soil. The results indicate that a mixture containing 6% emulsified asphalt and 9% of sand dunes effectively enhanced soil cohesion and friction angle [15].

Further studies have emphasized the role of silica fume in soil stabilization. Al-Obaidi et al. evaluated the effect of silica fume on the collapsibility and shear strength of gypseous soils. Their findings indicated that apparent cohesion increased by 140–310% in dry conditions and 20–40% in soaked conditions with the addition of 5–20% silica fume [16]. Similarly, Hayala et al. demonstrated that nano-silica significantly reduced the collapse potential of gypseous soil by up to 91%, highlighting its potential as an effective soil stabilizer [17]. Jassim et al. investigate how nano titanium dioxide affects the cohesion, and angle of internal friction of natural sandy soils with varying gypsum amounts. It is concluded that only small traces of nano titanium dioxide (0.5%) may be an advantageous agent for stabilizing collapsible gypseous soils [18]. Emad and Salman investigated the effect of nano clay and nano metakaolin on collapsibility and shear strength of gypseous soil with and without soaking. The collapse of soil is reduced, and strength is increased by increasing nano materials and time of treatment [19]. Abaas et al. show that calcium carbide residue and sulfuric acid can create a geopolymer which improve cohesion and collapse of gypseous soil [20].

Several alternative materials have been explored for soil stabilization. Hameed et al. investigated the combined effect of hydrated lime and waste plastic fiber on loose subgrade sand. They found that an 8% lime treatment provided the best strength improvements [21]. Jassam and Younes studied the impact of mixing sand dunes with 2–8% silica fume on gypseous soils, noting increased cohesion and internal friction angles under both soaked and non-soaked conditions. Their results also showed a decrease in collapse potential and an increase in unconfined compressive strength after seven days of treatment [22].

Another approach to soil stabilization involves the use of petroleum products. Hamad and Jassam investigated their effect on gypseous soils and found that increasing petroleum product content reduced collapse potential. However, they also observed that treatment efficiency diminished as gypsum content increased, suggesting that petroleum-based stabilization methods may be more

effective in soils with lower gypsum concentrations [23]. The effect of wetting on shear strength of gypseous soils was studied by Abd, it is found that the cohesion of soil is relatively good with the field water content, but it considerably reduced after soaking with water. When using kerosene for soaking gypseous soil, the friction angle of soil is increased [24].

The literature survey on gypseous soil stabilization demonstrates the significance of different materials and techniques to improve soil stability. A wide variety of lime products are used and are effective as stabilizers that can be combined with silica fume to improve the effectiveness of the stabilizer. Other stabilizers that have been investigated with success include chemical grouting, nano-silica and other waste plastics such as fiber.

Conventional stabilizers like lime or cement improve the mechanical properties of gypseous soils but are unable to offer permanent waterproofing, since they don't solve the problem of the solubility of gypsum. This research work is different from the traditional chemical stabilization method, in that the application of a hydrophobic barrier approach with KH-570 is used. It's all about moving the focus from structural binding to creating a water-repellent texture that impedes the dissolution process. It differs from traditional soil binder, which treats the surface energy of soil particles by eliminating the water from the soil surface, it was aimed at eliminating the cause of the collapse of gypseous soils, that is the water ingress. This new concept is a major contribution to sustainable geotechnical stabilization solutions, as it moves away from structural binding towards hydrophobic protection.

Silane compounds have demonstrated significant effectiveness as hydrophobic agents in various engineering applications. For instance, in concrete technology saline coupling agent improve interfacial bonding, reduce permeability, increase microhardness, and enhance mechanical durability [25-27]. Similarly, Silane treatments have been extensively employed in natural fiber/polymer composites to enhance fiber-matrix compatibility and improve mechanical properties [28]. Also, Silane-based coatings have emerged as environmentally friendly alternatives for corrosion protection of metals due to their strong adhesion and barrier performance [29]. Therefore, extending this approach to gypseous soils represents a logical and promising research direction. However, despite these established benefits in other fields, the specific effect of silane agents on mitigating gypsum dissolution and collapse behavior in gypseous soils has not yet been sufficiently investigated. This study provides an insight to the effect of such agents on important engineering parameters of soil.

2. Materials and Sampling

2.1. Soil Samples and Water Used

Disturbed soil samples with gypsum content of 45% were taken from Salah Al-Din governorate in Iraq at a depth of 1.5 meter below natural ground level. The samples were air-dried at room temperature under laboratory conditions, avoiding oven drying to prevent dehydration or alteration of the gypsum minerals. Once dry the soil was lightly hammered to break up the lumps without grinding particles, with a wooden mallet. Material was then passed through a No. 4 sieve (4.75 mm) to ensure it was a uniform sample for testing. This preparation procedure was particularly chosen to avoid excessive temperature or mechanical degradation of the gypseous soil's natural physicochemical properties, especially those related to the amount of gypsum in the soil. The particle size distribution curve of the soil is shown in Fig. 1, based on the Unified Soil Classification System (USCS), the soil is classified as SP. Standard Proctor tests were conducted to determine the compaction characteristics as shown in Fig. 2. physical and engineering properties of the natural soil are summarized in Table 1.

Chemical tests including total soluble salts, gypsum content, organic matter and pH value were conducted on the natural soil, and the results are shown in Table 2. The method that used to determine the gypsum content in soil involve dry the gypseous soil at a temperature of 45° C for 48 hours until its weight becomes constant and then continue dry the same specimen at 110° C for 24 hours [30]. The below equation is used to calculate the gypsum content.

$$Gypsum (\%) = \frac{W_{45^{\circ}C} - W_{110^{\circ}C}}{W_{45^{\circ}C}} (4.778) * 100\% \tag{1}$$

Where; $W_{45^{\circ}C}$: specimen weight at 45° C, $W_{110^{\circ}C}$: specimen weight at 110° C, 4.778: inverse ratio of molecular weight of water to the molecular weight of gypsum. Distilled water was used in all laboratory testing.

Table 1. Physical and engineering properties of the used soil

Properties	Description/Value	Standard
Specific gravity (G_s)	2.58	(ASTM D854-14)
Liquid limit	-	(ASTM D4318, 2017)
Plastic limit	NP	
Gravel (%)	6.9	(ASTM D422)
Sand (%)	90.4	
Fines (%)	2.7	
Coefficient of uniformity (CU)	7.7	
Coefficient of curvature (CC)	0.61	
Unified soil classification	SP	
Field unit weight (γ_{field}) (kN/m^3)	14.11	(ASTM D1556-15)
Field moisture content, (ω)%	6.9	
Maximum dry unit weight ($\gamma_{d,max}$) (kN/m^3)	16.62	
Optimum moisture content (OMC) %	10.4	(ASTM D698-91)

Table 2. Chemical properties of the used soil

Properties	Value
Total soluble salts (%)	51.3
Gypsum content (%)	45
Organic matter (%)	0.13
PH	6.8

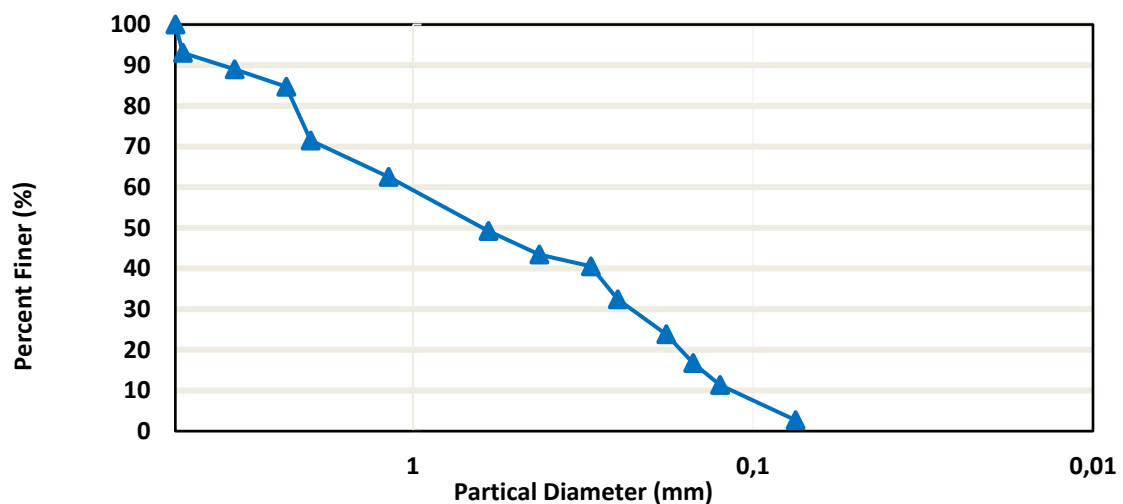


Fig. 1. Particle size distribution curve for the used soil

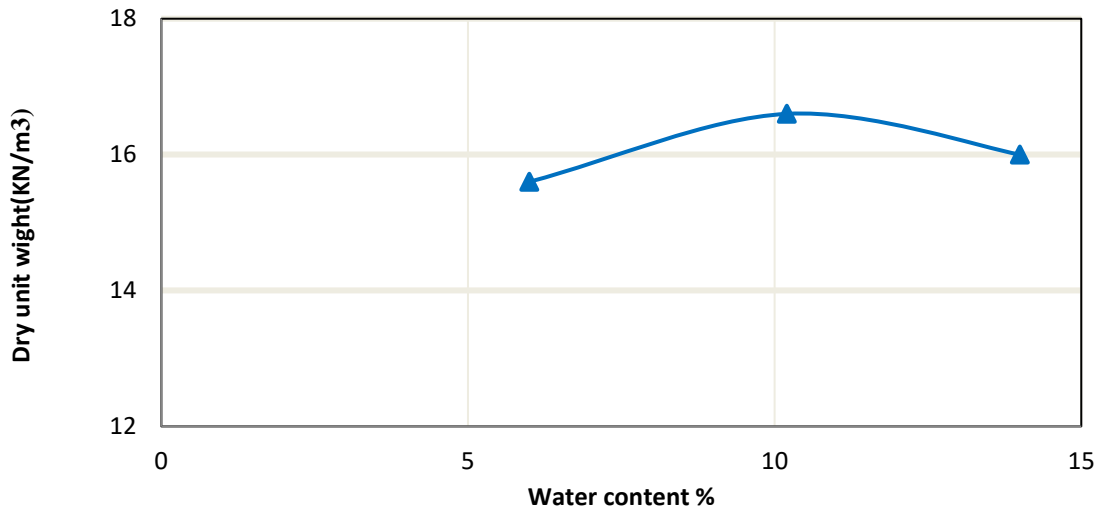


Fig. 2. Relation between dry unit weight and water content in compaction test for the used soil

2.2. Silane Coupling Agent

The silane agent used, 3-Methylacryloxy-Propyl Trimethoxy-Silane (also known as KH-570), is low viscosity organofunctional silane solution that can be added or sprayed on surfaces or texture to impart water repellency. Table 3 below shows the properties of the used silane coupling, this agent features a dual functional molecular structure consisting of a methacryloxy group and trimethoxysilyl groups. This molecular configuration is critical for creating the hydrophobic barrier, as the alkoxy groups hydrolyze and bond to the soil surface while the organic groups orient outward to repel water. All physical properties, including density and purity, were verified according to the manufacturers data.

Silane coupling agents have been introduced as promising materials for enhancing the waterproofing properties of surfaces. These agents may also react with the mineral parts of the textures and form a hydrophobic barrier, which decreases the water infiltration. However, the impact of silane coupling agents on the collapsibility and strength of gypseous soils remains a subject of ongoing research.

Table 3. Properties of the used silane coupling agent (kh-570)

Parameter	Description/Value
Appearance	Colorless clear liquid
Chromacity (Pt-Co)	10
Purity (%)	99.15
Boiling point	255°C
Density (ρ_{20} , g/cm ³)	1.048
Refractive Index (N_d , 25° C)	1.4285

In the current study, the silane agent is sprayed and mixed with soil samples. manual spraying device was used to ensure a gradual and uniform distribution over the soil surface. The spraying process was conducted in layers, where each portion of soil was sprayed separately and then soil was manually mixed for a duration of approximately (3–5 minutes) to achieve a homogeneous distribution of the agent. The mixing process was continued until no visible wet spots or untreated zones were observed.

To reduce the effect of environmental conditions on the effectiveness of silane agent, all sample preparation and treatment procedures were conducted under controlled laboratory conditions at a constant temperature of 25°C. This temperature was selected to represent typical ambient conditions and to ensure stability of the silane hydrolysis. Furthermore, after the application of the silane-based agent, the treated soil samples were allowed to cure for 24 hours under the same controlled temperature (25°C) and standard laboratory humidity conditions before any subsequent

testing. This curing period was essential to allow sufficient development of the silane bond and formation of the hydrophobic layer.

2.3. Preliminary Visual Test

An indicative visual test was performed to check the effectiveness of the waterproofing that can be provided by the utilized agent (KH-570) to the collected gypseous soil samples. As shown in Fig.3, four small approximately equal size soil clods prepared from disturbed samples were chosen with approximate weight is about 60 gr. per each sample; two for treated and two for untreated soil. The treatment in this test was done by spraying the agent solution as 4% weight of soil sample and absorbed by the surface of soil samples. It is important to note that this value was selected arbitrarily at this preliminary stage of the study, prior to conducting the full experimental program and before identifying the optimum value.

The treated soil samples were left for 24 hours for curing, then distilled water of 100 ml was added gently and simultaneously while they are in two separated beakers. The deformation due to soaking in water for each couple of samples are observed and recorded in different time intervals. A curing period of 24 hours is scientifically sufficient for the silane molecules (KH-570) to undergo hydrolysis and orient themselves onto the soil particle surfaces. The KH-570 agent is a surface energy modifying agent, unlike conventional stabilizers that need long-term curing to become chemically cemented. A protective hydrophobic matrix forms around the gypsum grains within 24 hours and offers instant protection against water dissolution. One of the benefits of using silane agents in geotechnical applications compared to traditional binders is the quick creation of the water repellent barrier. As shown in Fig. 3, while the untreated sample rapidly disintegrated (slaking) within the first few seconds, the sample treated with KH-570 retained its integrity even after 1 hour, this confirms the effectiveness of this material in protecting the soil and reducing gypsum dissolution.

2.4. Testing Procedures

2.4.1 Collapse Test

Collapse test (ASTM-D4546-23) is used to describe the collapse potential of a soil. The collapse potential can be determined by taking soil specimen at natural moisture content in a consolidation ring with a diameter of 63.5 mm and height of 20 mm. Step loads are applied to the specimen up to pressure level of 200 kN/m². At this pressure, the specimen is flooded for saturation and left for 24 hours. This test will provide the void ratios (e_1 and e_2) before and after flooding. The collapse potential (C_p) can then be calculated as:

$$C_p = \frac{e_1 - e_2}{1 + e_0} \quad (2)$$

Where; e_0 = normal void ratio of the soil, e_1 and e_2 are void ratios before and after soaking respectively. The foundation problems associated with a collapsible soil have been correlated with the collapse potential (CP).



(a): Time elapsed 1 sec.

(b): Time elapsed 3 sec.



(c): Time elapsed 7 sec.



(d): Time elapsed 30 sec.



(e): Time elapsed 10 min.



(f): Time elapsed one hr.

Fig. 3. Indicative visual test showing the waterproofing characteristic of the treated soil in comparison to untreated one. For each pair of beakers, the left side one is for treated sample

2.4.2 Direct Shear Test

The direct shear test (ASTM D3080/D3080M-11) was conducted to evaluate the shear strength parameters of the soil samples under controlled conditions. The test was performed using a standard shear box apparatus consisting of two horizontally split halves. The dimensions of box are (60x 60) mm. First, the soil sample was carefully prepared and placed inside the shear box at the maximum density and optimum moisture content. A porous stone was placed at the top and bottom of the specimen to allow drainage, followed by positioning a loading plate above the sample. A specified normal load was then applied vertically to the specimen and maintained constant throughout the test. After achieving equilibrium under the normal stress, a horizontal shear force was applied gradually by at a rate of 1 mm/min until the failure occurred. The test was repeated under different normal stresses of 100, 200 and 300 kPa.

3. Results and Discussion

3.1. Collapsibility Properties

The experimental results given show the collapsibility behavior of gypseous soil under applied vertical stress as shown in Fig. 4. The trends observed between the untreated soil and soil treated with various percentage (2%, 4%, and 6%) of the water repellent agent KH-570 give valuable insights into the effectiveness of the treatment. The untreated soil shows a large amount of reduction, especially after the soil is soaked with an applied stress of 200 kPa, suggesting a high collapse potential. This is a behavior typical of gypseous soil which, upon hydration, releases the gypsum and causes the soil to lose its structure.

With 2% KH-570 treatment, A significant decrease in collapse strain is observed with some evidence of collapsibility remaining. It looks as if the structure is resisting degradation due to hydration,

which indicates some improvement in the soil stability. This stability is thought to be due to the hydrophobic shield provided by the agent which protects the gypsum particles from the hydrolytic effect of water.

The collapse strain is increased at 4% then decreased again at 6% of KH-570. The effect of varying dosage (2%, 4% and 6%) on collapse strain may be attributed to surface encapsulation and particle lubrication. The silane agent at the 2% concentration can completely coat the gypsum particles, giving the initial protection against dissolution. Given this small strain increase of 4%, however, it can be attributed to a lubrication effect, due to the additional amount of silane that delays the complete stabilization of particles. At 6% dosage, the agent begins to partially fill the pores and form a continuous hydrophobic network. This structural reinforcement overrides the lubrication effect, resulting in the maximum resistance to collapse and the lowest strain values. From an economic and practical perspective, although the 6% dosage provides a slightly better or comparable improvement, the 2% dosage is considered optimal. This is because it achieves a significant reduction in collapse strain with substantially lower material consumption, making it a more cost-effective solution. especially when considering large scale field applications.

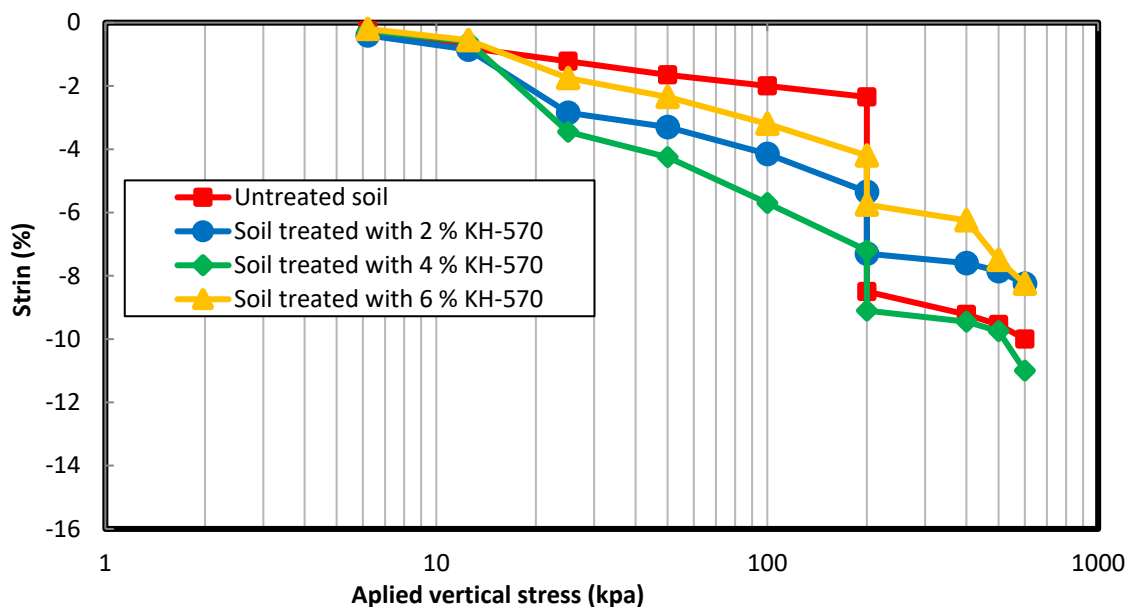


Fig. 4. Results of collapse test for untreated an treated soil with different percentages of KH-570

3.2. Shear Strength Parameters

Based on the previous section, it can be noted that the amount of silane coupling agent at 2% as weight ratio provides best results considering relative improvement and minimal amount of addition. This percentage is used in direct shear test for disturbed samples reconstituted with maximum density obtained from Proctor standard test. Four test scenarios are adopted when using the direct shear apparatus; first the samples were untreated and semi-dry, second was treated with 2% and semi-dry, third, untreated but soaked in distilled water for 24 hours before testing, and fourth was treated with 2% then soaked in distilled water for 24 hours before testing. Fig. 5 shows the results of direct shear test for these four cases; the results show a clear increase in both cohesion and angle of internal friction for treated soil in both semi- dry and soaked cases.

The results of soil cohesion and internal friction angle are shown also in Fig. 6 and Fig. 7 respectively including error bars representing the standard deviation. For the semi-dry case, adding 2% silane agent has improved both c and ϕ by 89% and 9.3% respectively. For soaked in distilled water for 24-hour case, taking the untreated and soaked case as a reference, these values were improved by 119%, 250% respectively. Although the value of ϕ that increase from 4° to 14° remains relatively low from a geotechnical standpoint, this enhancement indicates a meaningful improvement in shear resistance under soaked conditions, which is particularly relevant for gypseous soils known for their severe strength degradation upon wetting.

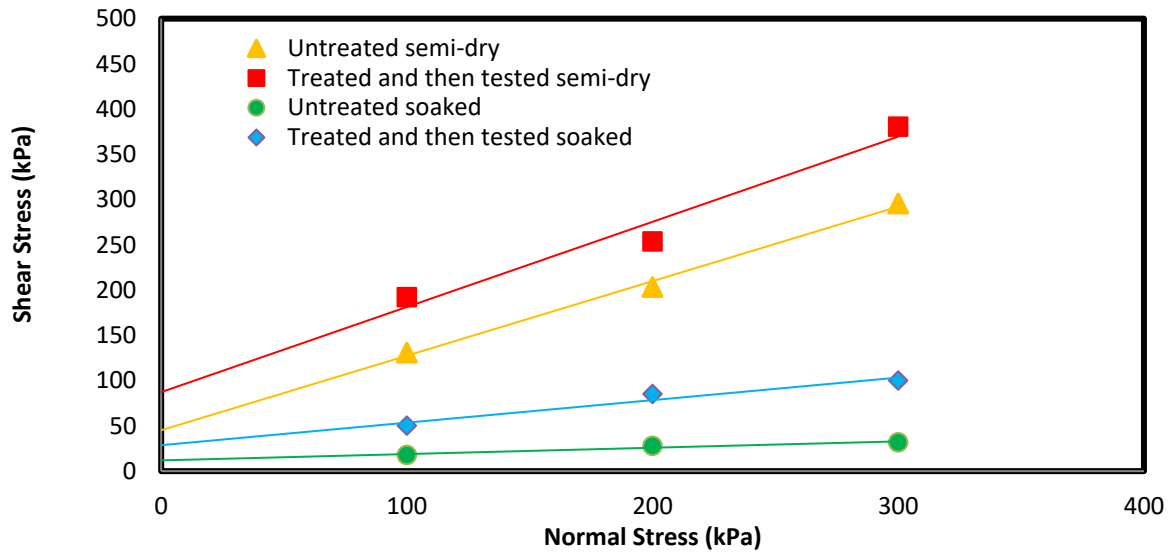


Fig. 5. Results of direct shear test on natural and treated soil

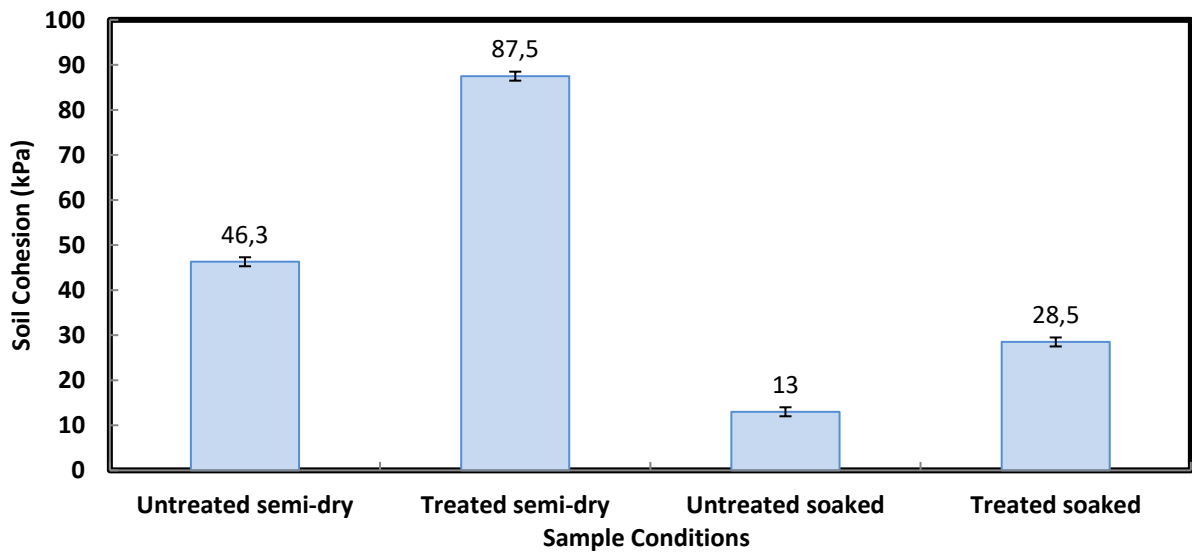


Fig. 6. Variation of soil cohesion under different conditions

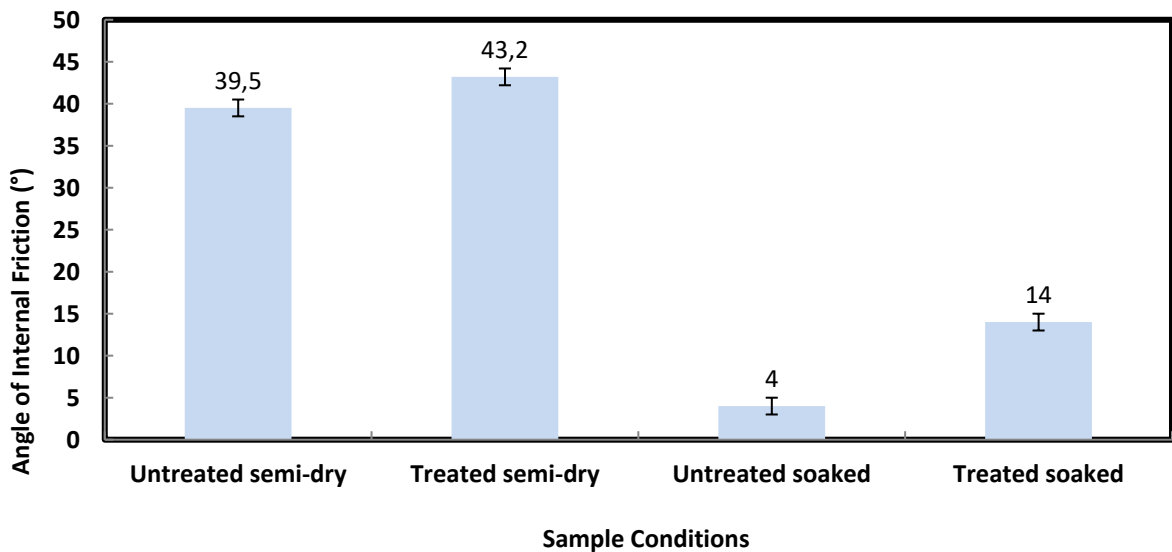


Fig. 7. Variation of angle of internal friction under different conditions

4. Conclusions

- It was found that the gypseous soil is highly prone to collapse when a load is applied on it, especially when soaked.
- Use of KH-570 as a hydrophobic treatment agent has proven to be an excellent method for reducing collapse potential of gypseous soil. KH-570 gives water-repellant properties to the matrix of soil, greatly reducing the amount of moisture that enters the soil and maintains interparticle cohesion. The results indicate that there is a systematic and consistent improvement in the mechanical response with the increase of KH-570 content, with the induced strain falling significantly and resistance to structural collapse increasing as KH-570 content increases.
- The treatment ratio of 6% provides the maximum improvement, but when engineering performance and material efficiency are considered, the treatment ratio of 2% KH-570 is found to be an optimal and practical solution. Significant improvements in stability and deformation control are obtained while using minimal amount of additives which provides a cost effective and practically viable stabilization strategy.
- The shear strength parameters were improved significantly with treatment: the cohesion of semi-dry case increased by 89%, the internal friction angle increased by 9.3%, while the c and ϕ of soaked case increased by 119% and 250% respectively when the optimum percentage of adding silane agent (2%) is applied. The results show that the silane agent is effective in improving the properties of the soil.
- Whereas silane-based materials like KH-570 have a relatively high cost per unit but they require a much smaller dosage as compared to other traditional stabilizers (like lime, cement etc.) that requires 5–10% by dry weight of soil. Further, the mode of action of KH-570 is quite different, and the ability to create a hydrophobic barrier helps to reduce the dissolution of the gypsum. It can lower the maintenance expenses and degradation in the long term, which are usually not considered in the material cost comparison. In this study, the (cost-effective) is a compromise between low dosage and high durability requirements as well as potential reductions of long-term damage, not just the initial cost of material.
- Future research is suggested to emphasize the durability of KH-570-treated gypseous soils, especially those in which degradation has been simulated to represent those occurring in the field. The laboratory scale validation done with a spraying and mixing process to provide controlled and homogenous application of KH-570 should be adapted in the field, e.g., surface spraying and mechanical mixing for shallow soil layers or subsurface treatment by chemical injection and jet grouting for deeper soil layers. Leaching effects and cyclic wetting–drying conditions are the most significant factors affecting the stability of gypseous soils and deserve special attention. Further studies were also needed to incorporate SEM, FTIR, and XRD analysis to study the changes in the microstructures and chemical interactions amongst the minerals of gypseous soil and the KH-570. In future studies, permeability tests are recommended to measure the effect of the KH-570 treatment on the decrease in water infiltration.

Acknowledgement

The authors would like to express their appreciation to the staff members of the soil mechanics laboratory, Civil Engineering Department, Tikrit University for their cooperation and help in accomplishing this paper.

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