Tikrit Journal of Engineering Sciences (2025); 32(4): 2319
DOI: http://doi.org/10.25130/tjes.32.4.24

IRAQI Academic Scientific Journals

ISSN: 1813-162X (Print); 2312-7589 (Online)

Tikrit Journal of Engineering Sciences

available online at: http://www.tj-es.com

TJES Tikrit Journal of Engineering Sciences

Effect of Waterproofed Nano-silica on Some Engineering Properties of Gypseous Soil

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Keywords:
Nano-silica; Waterproofed additive; Hydrophobic; Hydrophilic; Gypseous soil; Collapsibility.

Highlights:

- *Gypsum soils lose strength when wet due to gypsum bond dissolution.*
- *Hydrophobic nano-silica improves gypseous soil properties.*
- *It boosts cohesion and friction angle, even when the soil is submerged.*
- *It lowers collapsibility and water uptake better than hydrophilic nano-silica.*

ARTICLE INFO

Article history:

Received	06 Sep. 2024
Received in revised form	24 Oct. 2024
Accepted	06 Dec. 2024
Final Proofreading	26 Aug. 2025
Available online	30 Aug. 2025

Citation: Abd KG, Abd AH. **Effect of Waterproofed Nano-silica on Some Engineering Properties of Gypseous Soil**. *Tikrit Journal of Engineering Sciences* 2025; **32**(4): 2319.
http://doi.org/10.25130/tjes.32.4.24

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Abstract: Gypsum soils are prevalent worldwide, and their primary engineering challenge lies in the loss of mechanical properties upon exposure to water due to the dissolution of the gypsum bond. Many previous studies have explored various additives to improve this soil type; among these, nanomaterials have emerged as promising candidates for enhancing the geotechnical properties of soil. This study compares the effects of adding different proportions of regular (hydrophilic) nano-silica and waterproof (hydrophobic) nano-silica to gypseous soil with varying gypsum contents (13%, 30%, and 45%). Laboratory tests were conducted on both semi-dry and submerged disturbed samples, including direct shear, collapsibility, and water absorption tests. For the semi-dry case, and in comparison to untreated samples, the cohesion of hydrophilic and hydrophobic nano-silica-treated soil showed improvement, reaching up to 112% and 30%, respectively. The angle of internal friction has lower sensitivity to the above additives with increases of 11% and 10%, respectively. However, for the soaked state, the soil cohesion decreased with the increase in the hydrophilic nano-silica ratio, whereas it improved by up to 70% for the waterproofed one. Interestingly, the angle of internal friction reached 53% and 54%, respectively. Regarding the collapsibility coefficient and water absorption, the nano-silica-treated soil exhibited contrasting behavior. Hydrophilic nano-silica increased the collapsibility coefficient and water absorption with increasing addition, while waterproof nano-silica showed significant improvements, resulting in a reduction of 5% to 59% in the collapsibility coefficient and a decrease in water absorption. These results suggest using hydrophobic (waterproofed) nano-silica instead of hydrophilic one to improve the engineering characteristics of gypseous soil.

تأثير صغائر السيليكا المقاومة للماء على بعض الخواص الهندسية للتربة الجبسية

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الخلاصة
تنتشر التربة الجبسية في جميع أنحاء العالم، ويكمن التحدي الهندسي الأساسي لها في فقدان خواصها الميكانيكية عند تعرضها للماء بسبب ذوبان روابط الجبس. وقد استكشفت العديد من الدراسات السابقة إضافات مختلفة لتحسين هذا النوع من التربة؛ ومن بين هذه المواد، برزت المواد النانوية كمرشح واعد لتعزيز خواص التربة الجيوتقنية. تقارن هذه الدراسة تأثير إضافة نسب مختلفة من السيليكا النانوية العادية (الجاذبة للماء) والسيليكا النانوية المقاومة للماء (الطاردة للماء) إلى التربة الجبسية ذات المحتويات الجبسية المختلفة (13%, 30%, 45%) . أجريت الاختبارات المختبرية على العينات المشوشة شبه الجافة والمغمورة، بما في ذلك اختبارات القص المباشر والانهيار وامتصاص الماء. بالنسبة للحالة شبه الجافة وبالمقارنة مع العينات غير المعالجة، فإن تماسك التربة المعالجة بالنانو سيليكا الجاذبة للماء والطاردة للماء، أظهر تحسناً يصل إلى ١١٢٪ و٣٠٪ على التوالي. زاوية الاحتكاك الداخلي لديها حساسية أقل للمضافات المذكورة أعلاه بزيادة قدرها ١١٪ و١٠٪ على التوالي. أما بالنسبة لحالة النقع، انخفض تماسك التربة مع زيادة نسبة السيليكا النانوية (الجاذبة للماء)، بينما تحسن بنسبة تصل إلى ٧٠٪ للتربة المقاومة للماء، ومن المثير للاهتمام أن زاوية الاحتكاك الداخلي وصلت إلى ٥٣٪ و٥٤٪ على التوالي. فيما يتعلق بمعامل الانهيار وامتصاص الماء. أظهرت التربة المعالجة بالسيليكا النانوية سلوكاً متناقضاً؛ زادت النانو سيليكا الجاذبة للماء من معامل الانهيار وامتصاص الماء مع زيادة الإضافة، في حين أظهرت السيليكا النانوية المقاومة للماء تحسينات كبيرة، تتراوح من ٥٪ إلى ٥٩٪ انخفاضاً في معامل الانهيار وتقليل امتصاص الماء. تشير هذه النتائج إلى استخدام السيليكا النانوية الطاردة للماء (المقاومة للماء) بدلاً من السيليكا الجاذبة للماء لتحسين الخصائص الهندسية للتربة الجبسية.

الكلمات الدالة: نانو سيليكا، مقاوم للماء، طارد للماء، جاذب للماء، تربة جبسية، الانهيارية.

1.INTRODUCTION

There is a growing need to utilize peripheral areas for construction, including projects like city expansion and infrastructure development. However, some of these areas have weak soils that lack the necessary engineering properties. Consequently, it becomes imperative to improve and stabilize the soil to meet the engineering standards and transform it into a solid foundation for construction. The importance of improving and stabilizing foundation soils has been recognized since the inception of construction projects, and over time, new solutions and technologies have emerged [1]. Soil stabilization refers to the methods used to modify the texture of the soil and improve its geotechnical properties. These methods aim to increase shear strength, reduce permeability and compressibility, thereby enhancing the soil's bearing capacity. Generally, there are two types of soil stabilization: mechanical and chemical, as well as compaction [2]. Gypseous soils are found throughout the world, including in Iraq, where they account for approximately 31.7% of the nation's surface area. Due to the gypsum slates' susceptibility to leaching and dissolution by the action of water flowing through the soil mass, the presence of these soils, which can occasionally have considerable gypsum concentrations, presents challenging issues for buildings and strategic projects [3]. Gypsum in soil can dissolve upon exposure to water, resulting in increased soil compression and a decrease in its resistance to shear strength. As a result, gypseous soil is regarded as unstable soil because it may result in serious engineering issues, such as the collapse of projects built on it. In addition to the gypsum mass's surface area and the length of time it is exposed to water, the solubility of the gypsum depends on several variables, including the speed of the water's flow, its temperature, the concentration of salts and chemical compounds, and many more. By the wind carrying gypsum soils and blending them, gypsum can be created [4]. Conventional stabilizers, such as lime, cement, fly ash, rice husk ash, and even petroleum products, have been utilized in numerous projects. Literature such as the references [5-8] has extensively studied their effects. In recent years, there has been considerable interest in nanotechnology and nanomaterials [9-11]. The use of nanomaterials in soil enhancement was first explored in some earlier research. Regrettably, there has been little research conducted in this area regarding how the application of nanomaterials can improve undesirable geotechnical soil properties. Recently, many attempts have been made to treat problematic soil using nanomaterials for geotechnical and construction engineering applications, such as nano-silica, nano-alumina, and nano-cooper. The influence of nanomaterials on the chemical and geotechnical properties of soils is of great interest to investigate [12].

2.IMPROVEMENT OF SOIL BY NANOMATERIALS

Large amounts of soil are utilized in the field of civil engineering, particularly when constructing earth structures such as highways and embankments. For this reason, it is crucial to assess a soil's suitability while considering its strength, permeability, and compressibility [13, 14]. Our understanding and appreciation of nanotechnology have grown in all fields of knowledge over the past 15 years. However, geotechnical engineering has been at the forefront of nanoscale research for quite some time. Geotechnical engineers have been working with materials and phenomena at the nanoscale for a long time, long before nanotechnology became a mainstream topic [15]. Soft soils were stabilized using three types of nanomaterials: Nano-copper, Nano-clay, and Nano-magnesium. Small amounts of nanomaterials were added, not exceeding 2% of the soil's weight. The results showed a decrease in the plastic limit, liquid limit, plasticity index, and linear shrinkage. The dry density and compressive strength increased with the increase in the percentage of nanomaterials, and a decrease in the optimum moisture content was observed with the increase in the percentage of nanomaterials [16]. The effect of nano-silica and synthetic pozzolanic material on sandy soil was investigated by Ghasabkolaei and Choobbasti [17]. The findings suggested that the introduction of cement and nano-silica resulted in enhancements in the engineering properties of the sand. As the cement content increased, there was a noticeable rise in the maximum dry weight of the sand. Moreover, when suitable amounts of nano-silica were present, it significantly improved the mechanical characteristics of both the cement and the sand. Shokatabad [18] focused on the addition of Taftan natural pozzolan and nanomaterials, specifically nano-clay and nano-silica, to improve the stability of sandy soil. On the other hand, Sharma et al. [19], Al-Swaidani et al. [20], and Firoozi et al. [21] have used nanomaterials practically in expansive soils. Al-Obaidi et al. [22] investigated the effects of nanomaterials on collapsible soil behavior by applying two types of nanomaterials: nano-alumina and nano-silica. The results showed that using the optimum percentage of various nanomaterials reduced the potential for collapse. Mahdi et al. [23] investigated the effect of nano-clay materials on the collapsibility of disturbed gypseous soil samples. The test results indicated that mixing nano-clay with gypseous soils affected the consolidation behavior and hydraulic conductivity of the soils, regardless of the percentage of nano-clay. The collapse potential of soil samples mixed with 4% of nano-clay decreased by 77%. Generally, using 4% nano-clay produced the best improvement in the chemical and geotechnical properties of gypseous soils. Additionally, on the same side, Al-Gharrawi et al. [24] investigated the effect of nanomaterials, such as nano-clay and nano-silica, on the collapsibility of disturbed gypseous soil, utilizing these as very fine materials. The results showed that the addition of 1% of nano-silica can decrease the collapsibility by up to 91%. Almurshedi et al. [25] investigated the effect of nano-silica fume NSF on the collapsibility and shear strength of gypseous soil before and after soaking. Three percentages of microsilica fume, i.e., 1, 2, and 4%, were mixed with gypseous soil samples. By increasing the amount of nano-silica fume, the collapse potential of gypseous soil was lowered. Additionally, extending the curing period and adding more NSF led to an increase in soil shear strength. To summarize, several previous studies have utilized nanomaterials to enhance the properties of weak and unstable soils. It can be observed that nano-silica has been recommended by most studies dealing with gypseous soil. However, ordinary nano-silica may not be the best solution for gypseous soil, especially upon wetting, mainly because it naturally attracts water, which can weaken the gypsum bonds. The present study investigates the effect of treated nano-silica on the water repellency (hydrophobicity) of gypseous soil, specifically its impact on the shear strength and collapsibility. Additionally, the present study compares the effect of treated nano-silica with that of untreated nano-silica (hydrophilic).

3.EXPERIMENTAL STUDY

The work program includes three soil samples, each with a different gypsum content. The studied samples were treated by either hydrophilic nano-silica or waterproofed (hydrophobic) nano-silica for the purpose of improving their geotechnical properties. Additionally, laboratory tests, including collapse, direct shear, and water absorption tests, were conducted.

3.1.Soil Sampling and Properties

In this study, three types of disturbed natural gypseous soil were used. The samples were collected from three locations in the Salah al-Din Governorate. The samples were taken at depths ranging from 0.5 to 1 m below the natural ground surface. The first one was obtained from Tikrit City, with a gypsum content of 13% named hereafter (Soil 1), the second soil sample was extracted from Baiji City with 30.26% gypsum content (Soil 2). The third was taken from Tikrit University's main campus, which has a high gypsum content of 45.8% and is designated as (Soil 3). Figure 1 shows a map of locations from which the above three samples were collected. Figure 2 shows the grain-size distribution curve. Table 1 presents the results of the physical properties and chemical tests conducted on the three soil samples studied.

3.2.Nano-Silica

Two types of nano-silica were used in this study: Untreated nano-silica (hydrophilic) and treated nano-silica to be waterproofed (hydrophobic). Both are imported from China, and their properties are listed in Table 2. Additionally, Fig. 3 illustrates the size distribution of the silica nanoparticles. For both types of nano-silica, three ratios (1%, 2%, and 3% by weight of dry soil) were added and mixed with the soil samples.

Fig. 1 Image of the Samples' Locations on the Map of Iraq.

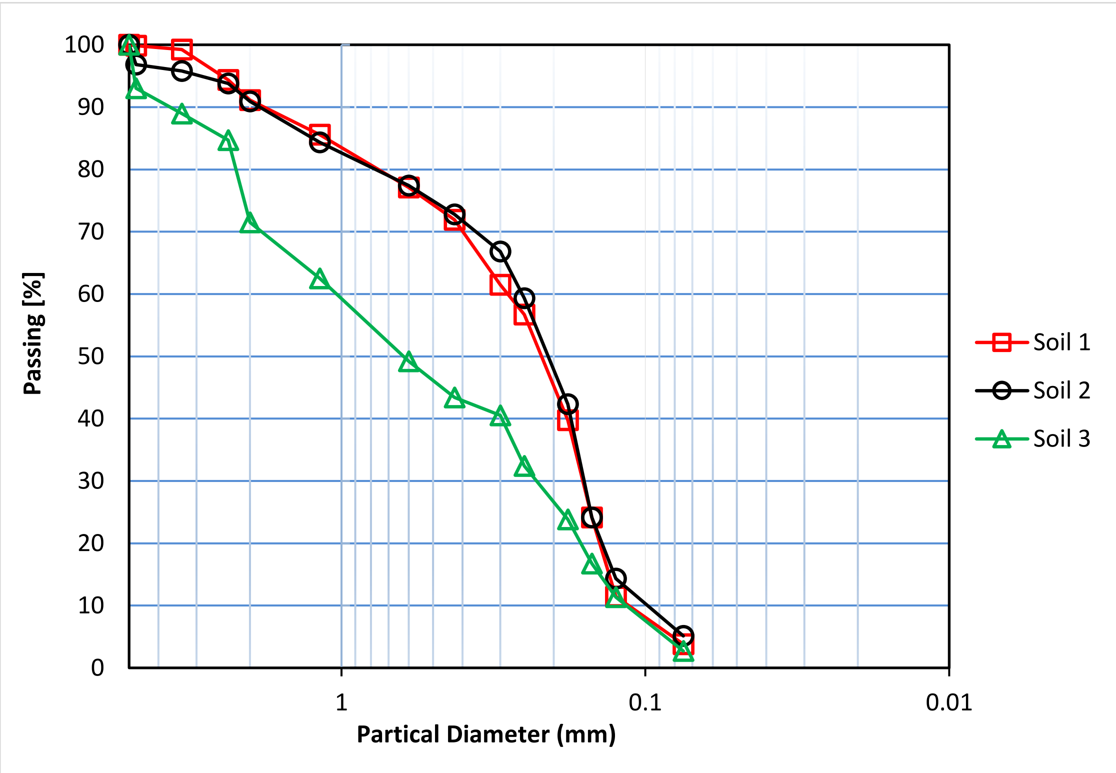

Fig. 2 The Grain-Size Distribution Curve.

Table 1 Physical and Chemical Properties of Natural Soils.

Properties		Soil 1	Soil 2	Soil 3	Standards
Field moisture content (ω)%		5.1	4.02	6.4	(ASTM D1556,15)
Specific gravity (Gs)		2.51	2.34	2.25	(ASTM D854-14)
Atterberg Limits	Liquid limit (L.L)%	-	-	-	(ASTM D4318, 2017)
	Plastic limit (P.L)%	N.P	N.P	N.P	
Unified soil classification system		SP	SP – SM	SP	(ASTM D422)
Sand %		92.99	94.72	90.4	
Fines %		3.8	5.13	2.7	
Coefficient of uniformity (C_u)		2	2	7.8	
Coefficient of curvature (C_c)		2.07	2	0.612	
Field unit weight (γfield) KN/m³,		14.88	14.3	13.81	(ASTM D1556, 15)
Compaction Test (standard method)	Maximum dry unit weight (γmax) (KN/m³)	18.32	16.89	16.62	(ASTM D698-91)
	Optimum moisture content % (O.M.C)	10.8	8.25	10.5	(ASTM D698-91)
Organic matters (O.M)%		0.3879	0.5172	0.013	IS 2720 22
pH value		7.47	7.32	2.28	Iraqi Standard Specification Modified 2003
Gypsum Content %	1st method	14.93	36.8	44.3	US Salinity Laboratory Staff, Richards (1954)
	2nd method	13.02	30.26	45.8	(A. Al-Mufti and Nashat, 2000)

Table 2 The properties of nano-silica (waterproofed) and nano-silica (hydrophilic) as given by the Simel Chemical Industry Co., Ltd.

Properties	Nano-silica Hydrophilic	Nano-silica (waterproofed)
Appearance	White	White
Silicon Oxide Content	99%	98.1%
Particle Size Distribution (D 50)	20nm	20 nm
Specific Surface Area	172 m2 /g	134 m2 /g
Density	0.04 g/cm3	0.04 g/cm3
Moisture	2.7 %	2.7 %
Loss on ignition	5.2 %	5.2 %
Surface treatment	None	Hydrophobic
pH	5.5 – 6	5.5 – 6

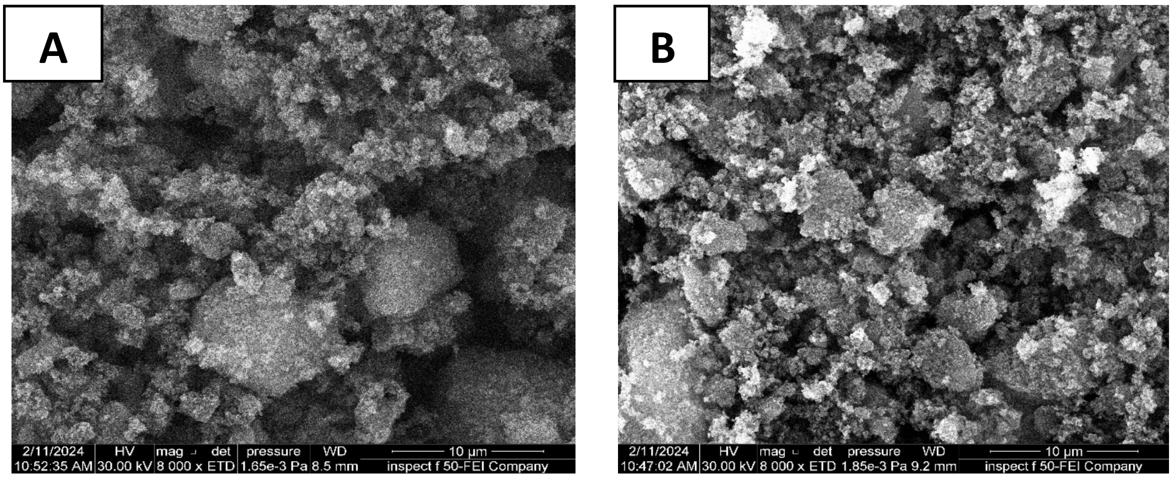

Fig. 3 SEM Images for the Used Nano-Silica (A) Hydrophilic, (B) Hydrophobic (Waterproofed).

4.RESULTS AND DISCUSSIONS

4.1.Shear Strength Parameters

A series of direct shear tests was conducted to determine the shear strength parameters for natural (untreated) and gypsum soils treated with either hydrophilic or hydrophobic (waterproofed) nano-silica. Tests were performed based on the procedure suggested by ASTM D3080 [26]. Two cases were considered: semi-dry samples and submerged (soaked) samples for 24 hours. Three normal pressures, i.e., 100, 200, and 300 kPa, were applied to determine the stress-strain relationship for the tested soil samples. The results of soil cohesion and angle of internal friction are listed in Table 3.

Table 3 The Results of the Direct Shear Test on Natural Soils.

Parameter	Soil 1	Soil 2	Soil 3
Apparent cohesion (c) (kPa)	14.56	26.81	46.32
Apparent cohesion (c) after soaking (kPa)	7.51	9.93	12.96
Friction angle (ϕ) (degrees)	31.59	32.38	39.48
Friction angle (ϕ) after soaking (degrees)	3.01	2.98	4

4.1.1.Apparent Cohesion

For the semi-dry case, the apparent soil cohesion seems to improve for both hydrophilic and waterproofed nano-silica-treated soil, as shown in Fig. 4. Actually, the hydrophilic nano-silica yields better results that can reach up to 112% of the cohesion of untreated soil, corresponding to a 2% weighted ratio of hydrophilic nano-silica. While the best improvement using waterproofed nano-silica was 30%, corresponding to a 1% addition of nano-silica. However, for the soaked case, the hydrophilic nano-silica had a negative impact on soil cohesion, as can be seen in Fig. 4. This behavior may be attributed to the attraction of water molecules, allowing water to flow among soil particles and thereby increasing the solubility of gypseous bonds. On the other hand, the waterproofed nano-silica provided an improvement of up to 70% compared to the untreated soaked soil. The best waterproofed nano-silica ratio here was still 1%. This behavior may be attributed to the fact that the waterproofed nano-silica particles act as barriers, reducing water infiltration into the soil and thereby reducing the interaction between water and gypsum.

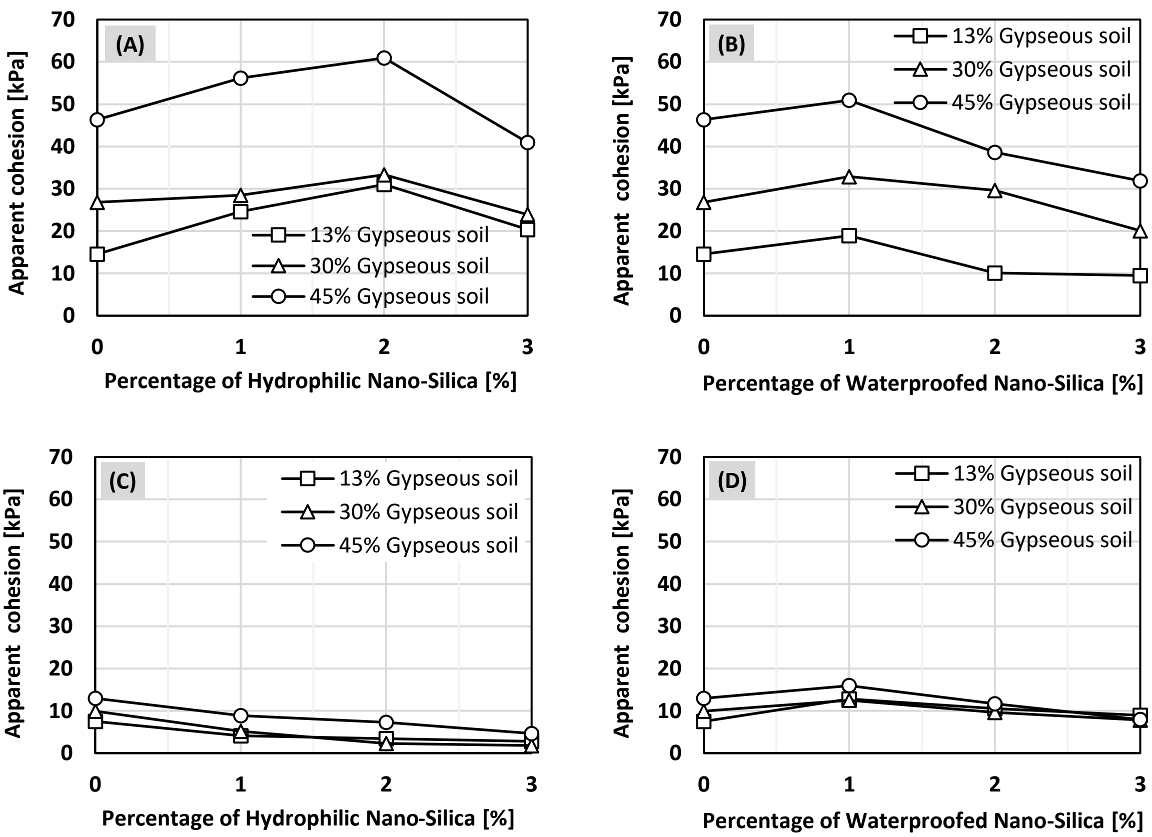

Fig. 4 Variation of *apparent* cohesion comparing the effect of adding hydrophilic and hydrophobic (waterproofed) nano silica for different cases. (A) and (B) for the semi-dry case, and (C) and (D) for the soaked case.

4.1.2.Angle of Internal Friction

The angle of internal friction was determined from a direct shear test after preparing the samples and storing them in plastic bags for 24 hours to allow for curing. Figure 5 summarizes the relationship between the angle of internal friction value and the percentages of nano-silica in semi-dry and soaked conditions. It can be seen that the internal friction angle was much higher in the semi-dry state than in the soaked state. For the semi-dry case, the results in Fig. 5 showed an increase in the value of the angle of internal friction when using hydrophilic nano-silica and when using the waterproofed nano-silica by (11% and 10%) with the ratio of (2% and 1%), respectively. The nano-silica powder is very fine and may not significantly contribute to the friction behavior; consequently, no significant improvement was observed in the angle of internal friction. In the soaked case, the soil samples were submerged for 24 hours and then sheared while submerged to simulate the worst-case scenario that could happen in the field. A significant drop in friction can be noticed upon wetting; however, relatively, the soil with 1% hydrophilic nano-silica showed the best results, i.e., a 21% improvement in frictional angle compared to untreated soaked soil. This improvement was doubled (42%) when utilizing 1% of nano-silica that had been waterproofed.

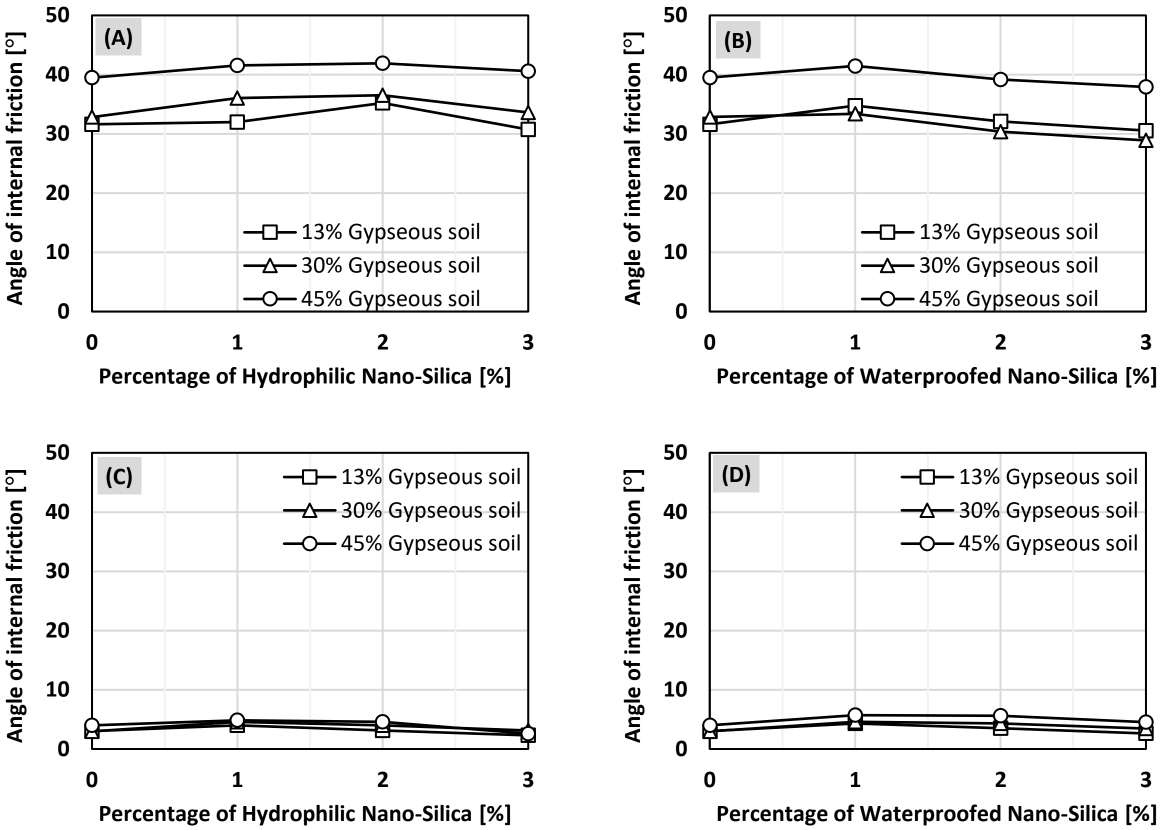

Fig. 5 Variation of the angle of internal friction (ϕ) comparing the effect of adding hydrophilic and hydrophobic (waterproofed) nano-silica for different cases. (A) and (B) for the semi-dry case, and (C) and (D) for the soaked case.

4.2. One-Dimensional Swell/Collapse Test

Any saturated soil that experiences a significant loss of volume and a fundamental rearrangement of particles as a result of saturation, with or without loading, can be considered a metastable soil [27]. To determine the soil's collapse strain (Ɛc), one-dimensional soil swell or collapse was conducted. Since the specimens were reconstituted, Test Method A was adapted as mentioned in ASTM D4546-23 [28]. When the specimen was ready for testing, it was placed in the loading device, and a seating stress of 1 kPa was applied, represented by the weight of the load plate and the top porous stone. Four soil samples were prepared with different stress levels achieved by applying vertical forces in increments. For every specimen, the following applied stresses were used, i.e., 6.25, 12.5, 25, 50, 100, 200, 300, 400, 500, and 600 kPa. The force on each specimen was raised in intervals of 5 to 10 minutes, with a maximum loading interval of one hour, to prevent the specimen from drying out. Each specimen was soaked in distilled water for 24 hours after the dial gauge readings representing the amount of compression, or h_1, were recorded. This procedure enables the measurement of the wetting-induced collapse, Δh_2. Being aware that the applied vertical stress for the first specimen was 50 kPa, while the applied stresses for the second, third, and fourth specimens were 100, 200, and 400 kPa, respectively. The collapse strain Ɛc (%) for the one-dimensional collapse test was calculated as follows:

$$\textbf{Collapse strain } \varepsilon c\ (\%) = -\frac{100\,\Delta h2}{h1} \quad (1)$$

where $h1$ is the specimen height immediately prior to wetting, $\Delta h2$ is the change in specimen height, and collapse caused by wetting.
The first sample was inundated at 50 kPa, and the second, third, and fourth samples were inundated at pressures of 100, 200, and 400

kPa, respectively. Figures 6, 7, 8, and 9 summarize the results of the (21) test samples in brief. Taking a quick look at the above-mentioned figures, one can notice two trends: first, the hydrophilic nano-silica increased the collapsibility in all cases, while the waterproofed nano-silica decreased it. Second, the higher the applied stress, the better the results of waterproofed nano-silica. The average improvement in the collapse strain corresponding to 50, 100, 200, and 400 kPa was 34%, 46%, 59%, and 49%, respectively. These values corresponded to 2% of waterproofed nano-silica.

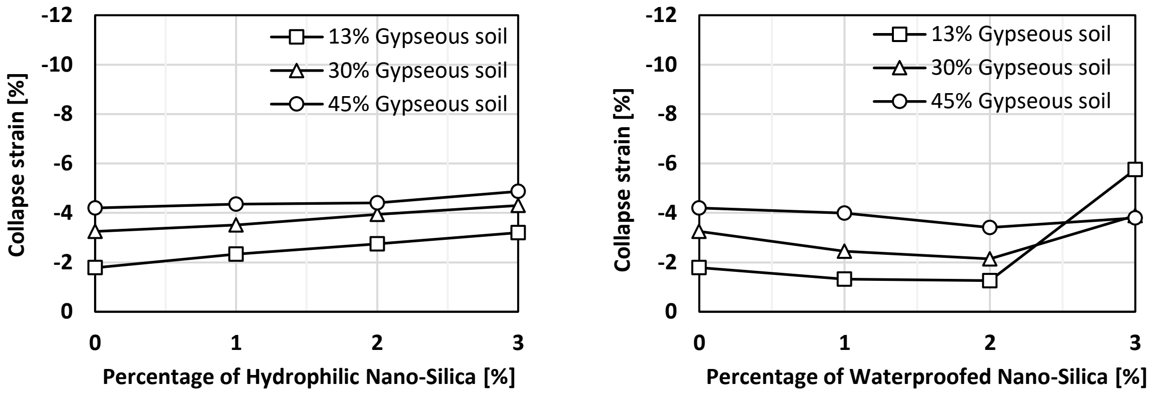

Fig. 6 Collapse Strain for 50kPa Comparing Soil Treated with Hydrophilic and Hydrophobic (Waterproofed) Nano-Silica.

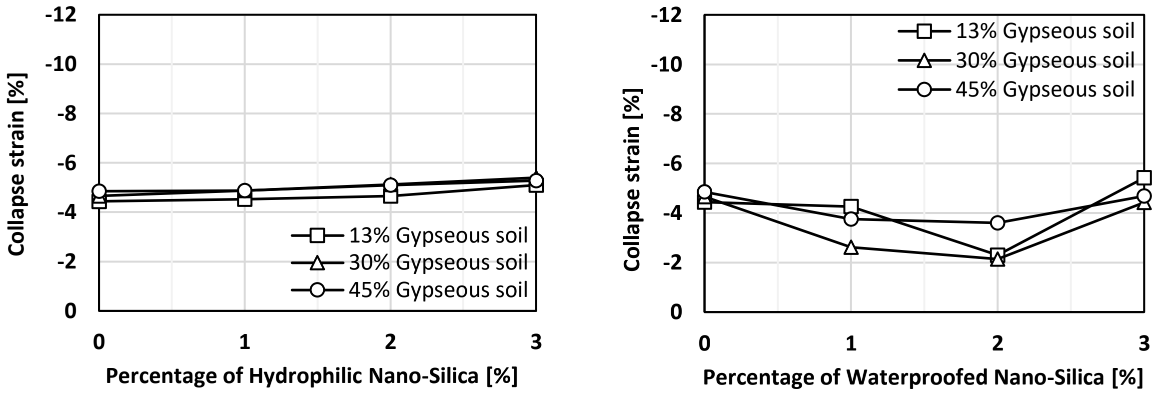

Fig. 7 Collapse Strain for 100kPa Comparing Soil Treated with Hydrophilic and Hydrophobic (Waterproofed) Nano-Silica.

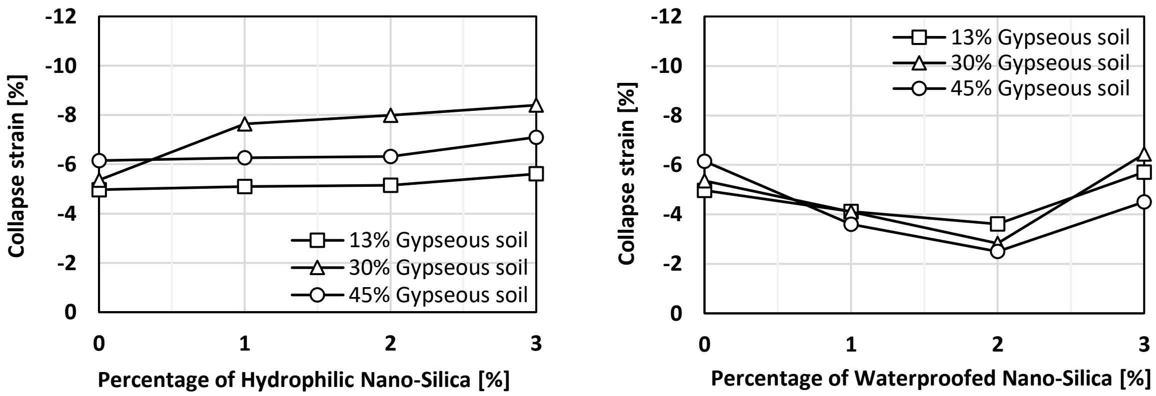

Fig. 8 Collapse Strain for 200kPa Comparing Soil Treated with Hydrophilic and Hydrophobic (Waterproofed) Nano-Silica.

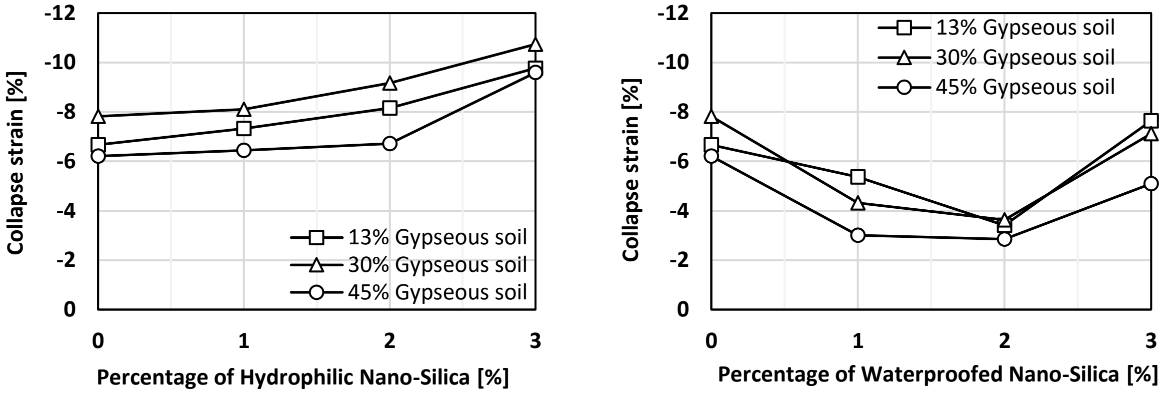

Fig. 9 Collapse Strain for 400kPa Comparing Soil Treated with Hydrophilic and Hydrophobic (Waterproofed) Nano-Silica.

4.3. Water Absorption

The water absorption is represented by the moisture content of the sample after it is fully saturated. The test was conducted in accordance with (ASTM D2216, 19) [29]. The moisture content was calculated from the relationship:

$$\omega\ \% = \frac{W2-W1}{W1} \qquad \textbf{(2)}$$

where ω % is the moisture content, *W1* is the wet soil weight(g), and *W2* is the dry soil weight(g).

The moisture content of untreated and nano-silica-treated soil samples after being submerged in water can indicate water infiltration, and consequently, gypsum solubility. Figure 10 compares soil mixed with hydrophilic and hydrophobic (waterproofed) nano-silica in terms of water adsorption. This comparison can also be used to measure the effectiveness of waterproofed nano silica. The hydrophilic nano-silica attracts water, thereby providing a proportional increase in water adsorption. Waterproofed nano-silica acts as a barrier, preventing water from percolating into the soil structure. As can be shown in Fig. 10, the average improvement in water adsorption was approximately 59% compared to untreated soil.

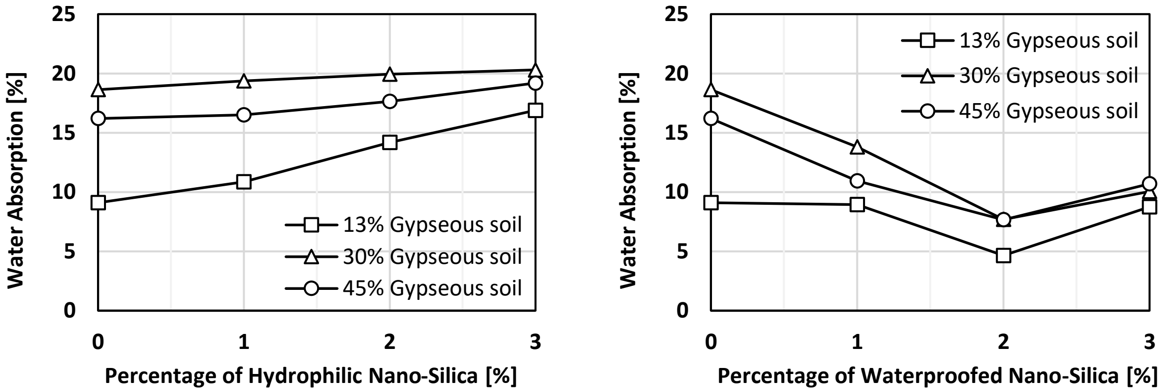

Fig. 10 Comparison of Water Absorption between Soil Mixed with Hydrophilic and Hydrophobic (Waterproofed) Nano-Silica.

5. CONCLUSIONS

The present study investigated the effect of adding nano-silica on the shear strength parameters, collapse strain, and water absorbability for gypseous soil. Additionally, it compared the effects of hydrophilic and waterproofed nano-silica in both dry and soaked conditions. Based on the results, the following points can be drawn as conclusions:

1) Hydrophilic nano-silica can improve the soil cohesion for gypseous soil only in semi-dry cases. In a soaked case, it had a negative impact. However, the waterproofed nano-silica can enhance soil cohesion in both cases up to 30% and 70%, respectively.
2) The angle of internal friction can be improved by mixing hydrophilic nano-silica in both semi-dry and soaked cases up to 11% and 10%, respectively. Interestingly, these ratios changed to 21% and 42%, respectively, when utilizing waterproofed nano-silica.
3) The average improvement of the collapse strain corresponding to 50, 100, 200, and 400 kPa was 34%, 46%, 59%, and 49%, respectively. These values corresponded to 2% of waterproofed nano-silica.

4) Soil absorption rate for water decreased by 59% for the soil treated by waterproofed nano-silica (hydrophobic).
5) The optimum ratio of waterproofed nano-silica that showed the best overall results, i.e., 1.5 %.
6) After comparing the two studied types of nano-silica, the waterproofed nano-silica was found to be more efficient, which is true for both semi-dry and soaked cases.

ACKNOWLEDGEMENTS

The authors are grateful for the staff support of the Civil Engineering Department, College of Engineering, Tikrit University. Postgraduate Research No. TU. G/2022/(5073-7-3).

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