E3S Web of Conferences **427**, 03005 (2023) https://doi.org/10.1051/e3sconf/202342703005
ICGEE 2023

Improving Engineering Properties of Soil for Highways Purposes by Halloysite Nanotubes

Itimad A. Mukheef [1, a*], Ibtehaj T. Jawad[1, b] and Abdul Rudha I. A. Al–Kremy[1]

[1]Department of Civil Engineering, University of Babylon, Babylon, Iraq

[a]itimadmu@gmail.com and [b]Eng.Ibtehaj.taha@uobabylon.edu.iq

*Corresponding author

Abstract. This study investigates the stabilizing effects of Halloysite nanotubes on the geotechnical features of gypseous soil as a subgrade. The soil that was utilized for this study was collected from Ayn al-Tamr in Karbala City, southwest of Iraq, having an average gypsum of 30% and was designated as (SP) by (USCS). As a percentage of the dry unit weight of the soil, the gypseous soil was mixed with a small amount of nanomaterial (Halloysite nanotubes). A set of physical and chemical identification tests have been conducted on the original soil, as well as additive, direct shear, collapsibility, and California bearing ratio (CBR) testing on both untreated and treated soil samples using Halloysite nanotubes. Three different additive proportions (2.5, 5, and 7.5) % by dry unit weight of soil were added to soil samples. The results of the conducted tests revealed that the geotechnical properties of the soil sample were considerably modified. As the amount of utilized nanomaterials increases, the collapse potential instantly decreases. In addition, soil strength and stability were increased by increasing CBR values, and the collapse severity was changed from moderate trouble to no problem. Thus, the value of 2.5% of Halloysite nanotubes can be considered as an optimum percentage based on the results of the mentioned tests.

Keywords: Halloysite Nanotubes; soil stabilization; gypseous soils; subgrade; engineering properties.

1. INTRODUCTION

There is considerable pavement cracking and early service loss on many small- to medium-sized roads constructed on gypseous soils, particularly in Iraq's western and northern regions, such as the provinces of Anbar and Salah al-Din. This is due to volume changes brought on by seasonal moisture content fluctuations clogging up roads constructed on problematic gypsum soils (such as substratum soils) [1]. The properties of the soil are significantly altered during soil stabilization to generate long-term strength and stability. Depending on the kind of pavement, the underlayment layer, which lies beneath the underlying course, is the lowest in pavement construction. The substrate typically includes a variety of locally accessible soil components, some of which may be too soft, damp, or both to be strong or rigid enough to support the loading of the pavement [2]. Therefore, to increase the performance of existing substratum materials, pavement design should concentrate on making the most effective, economical, and efficient use of those materials. The necessary treatment is needed to make loose and/or wet (weak) soils workable and compliant with technical standards needed for paving and other engineering construction works. For the pavement to be durable and perform economically, it is essential to understand the engineering characteristics of the substratum [3].

Due to its unpredictable behavior when water exposure, gypsiferous soil has not been utilized as a building material frequently. Gypsum ($CaSO_4.2H_2O$), the significant component of gypsiferous soil, is present [4-7]. According to [10], the term "gypsiferous soil" refers to soils that include more than 2% gypsum, while [11] defines gypsiferous soil which consists of at least 6% gypsum. In Iraq, [10] it has been found that gypsum concentrations of 3–10% do not appreciably affect soils' structure, consistency, or water-holding capacity. When gypsum content is sufficient to alter the soil's characteristics, soil can be referred to as "gypsum soil" in the field of civil engineering [11]. Gypseous soils are among the most complicated materials for geotechnical engineers. Gypseous soils are well known to have a high carrying capacity and very low compressibility when dry. It has been observed that gypseous soils can abruptly collapse when exposed to water. So, direct contact with water determines the collapsibility of gypseous soils [12]. In Iraq, the biggest issue with gypseous soils is exposure to seeping water. Moving water (unsaturated with gypsum) has been found to cause gypsum leaching and significant cavities. The rise in the groundwater table causes significant softening, leading to shear strength loss and increased settlement. During the lifespan of the road and structure, moisture or saturation of gypsiferous soil could result in a sudden settlement via collapse [13]. Because the soil's engineering properties are of poor quality, soil stabilization is currently a necessary activity. Changing the properties of soil to improve its performance for engineering and building applications is known as soil stabilization [13].

On the other hand, due to the innovative potential applications of nanoscale particles, nanotechnology has recently received significant scientific interest. Compared to materials with normal grain sizes and the same chemical composition, nanoscale materials can have considerably better characteristics [14]. Collapse potential (CP) decreased as an additional additive nano-clay was applied to the soil sample. The nano-clay has been applied to the soil in three different amounts (2.5, 5, and 10%). The additive alters the soil in a way that has no negative effects because it causes a decline in CP at a value of (73.75%) when nano-clay is present

[15,16]**.** Soil stabilization is just one of the many geotechnical applications in engineering that use nanotechnology and nanomaterials [17]**.**

2. MATERIALS

2.1 Gypseous Soil

The gypseous soil samples have been collected from Ayn al-Tamr (a city in central Iraq about 67 km west of Karbala province, nearby Razzaza Lake). From 1 m to 1.5 m below normal ground level. The samples are packed in bags and delivered to the soil mechanics laboratory at the University of Babylon in Babil province to be investigated. The characteristics of the selected soil are illustrated in Table 1.

Table 1: The features of gypseous soil.

Soil Property	Testing Standard	Gypseous Soil
Liquid- Limit (L.L), %	ASTM D4318	N.P
Plastic- Limit (P.L), %		N.P
Plasticity -Index (P.I), %		N.P
Clay % + Silt, %	ASTM D422	1.81
Sand, %		96.25
Gravel, %		1.94
Unified Soil Classification System (USCS)		SP
Specific- Gravity (Gs)	ASTM D854	2.61
Maximum Dry Density (MDD), gm/cm^3	ASTM D698	1.810
Optimum Water Content (OWC), %		12.60
Cohesion-(C), kPa	ASTM D3080	7.2
Angle of Internal Frication (ϕ)		40.81
Unsoaked California Bearing Ratio (CBR), %	ASTM D1883	9.85
Soaked California Bearing Ratio (CBR), %		6.08
Collapse Potential (C.P), %	ASTM D5333	3.42
Gypsum Content, %	BS1377:1990 Part3	30
Sulphate Salt (SO_3) Content, %		13.93

2.2 Halloysite Nanotubes

The naturally occurring tubular clay mineral halloysite ($Al_2Si_2O_5(OH)_4.2H_2O$) is a hydrated polymorph of kaolinite [17]**.** Halloysite nanotubes were provided by (Vas Chem Company- China) in the amount of 10kg. Table 2 lists the major Halloysite nanotube oxides identified through XRF analysis**.** Previous research concerning the application of nanomaterials in the stabilization of soil has found such materials to be commonly used in amounts ranging from 1% to 10% by weight of soil [18]. Therefore, (2.5, 5, and 7.5) % of Halloysite is chosen to select a comparable range and limit the number of tests.

Table 2: Major oxides of nanotubes of Halloysite.

Element	Atomic, %	Atomic % error	Weight, %	Weight % error
C	10.6	0.2	7.0	0.1
O	68.1	0.3	60.0	0.3
Na	1.2	0.0	1.5	0.0
Al	10.5	0.0	15.6	0.1
Si	6.6	0.0	10.2	0.1
S	2.7	0.0	4.8	0.0
K	0.3	0.0	0.7	0.0
Ca	0.1	0.0	0.2	0.0

2.3 Kerosene

Since gypsum is dissolvable material in water, kerosene was used in the specific gravity test for gypseous soil. The used kerosene has a density of 0.8 gm/cm$^{3.}$

2.4 Water

Distilled water was used to soak the gypseous soil in the collapse test. For other tests and curing, tap water has been used.

3. TESTS AND METHODOLOGY

3.1 Gypsum Content

This study measures the gypsum content of normal soil samples and soil samples treated with 2.5% Halloysite Nanotubes. The gypsum content can be calculated according to [19] to the sulfate content of the soil using Equation 1:

Gypsum (%) = $SO_3\ (\%) \times 2.15$ (1)

3.2 Specific Gravity

To overcome the problem of gypsum dissolution**,** [20] suggested using kerosene instead of distilled water, and this requires knowing the density of the liquid used at the laboratory temperature. The specific gravity was determined according to ASTM D854.

3.3 Grain Size Distribution

The dry sieving process was used by placing the soil in a group of sieves (4, 8, 20, 40,100, and 200), with a pan at the bottom and a cover at the top. The weights were taken by filling the empty sieve with soil and then calculating the remainder ratio according to ASTM D422.

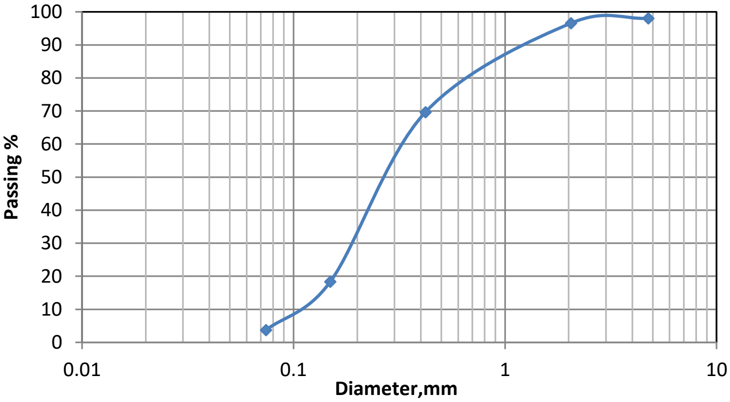

Figure 1: The soil's gypseous grain size distribution curve.

3.4 Atterberg Limits

Gypseous soil is nonplastic soil.

3.5 Compaction Test

According to ASTM D698, the original soil (untreated soil) and soil-Halloysite nanotubes (treated soil) mixtures have been exposed to standard proctor compaction tests at different proportions of additive (2.5, 5, and 7.5)% and with distinct quantities of water (3% cumulative).

3.6 Direct Shear-Test

The direct Shear test was conducted to determine the soil samples' shear strength parameters. The size of the sample was 60cm X 60 cm X 40 cm at the respective MDD and OMC state of soil for treated and untreated samples. The tests were conducted at vertical normal stresses of 50, 100, and 200 kPa.

3.7 Collapsibility Test-Single Collapse-Test

In order to determine the soils' collapsing potential (CP), just a single oedometer test has been performed. The samples were gently tamped in a compaction mold to prepare them at the MDD and OWC. A collapse test sample was conducted by pressing the odometer ring to the soil. Put the sample ring, filter paper, and porous stones as quickly as possible. The soil sample uses a sitting pressure of 5 kPa for a single odometer. Within 5 minutes of applying the seating stress, we increase the load every hour at normal water content up to the appropriate vertical stress (200 kPa). Stress increments should be (25, 50, 100, 200) kPa. We record the dial gauge reading before each load increase. After that, it is soaked in distilled water for one day, and the two heights determine the difference before and after immersion. According to [4], the CP can be determined by using Equation 2:

$$\mathbf{C_P} = \frac{\Delta e}{\mathbf{1+eo}} 100 \tag{2}$$

3.8 California Bearing Ratio Test (CBR)

According to ASTM D1883, the CBR test is conducted in a lab on the soil sample after delivering it to the required compaction ratio, when it has an MDD and OWC. Samples (soaked and unsoaked) are prepared for CBR examination in cylindrical metal molds (the mold sizes, according to AASHTO, are 6 inches in diameter and 7 inches in height). Four soil specimens (soaked and unsoaked) with Halloysite were dry mixed for each ratio (0, 2.5, 5, and 7.5) % and then compacted in the mold at MDD and OWC according to the standard proctor test. The soaked samples were put in a tank of water for 96 hours to calculate the swelling and CBR ratio for each ratio of Halloysite nanotubes.

4. RESULTS AND DISCUSSION

4.1 The Effect of Halloysite Nanotubes on Soil Compaction Parameters

The compaction curves from standard-Procter are shown in Figure 2. Tables 3 and Figures 3 and 4 illustrate the optimum moisture content (OMC) and maximum dry density (MDD) for all soil mixtures (0.2.5, 5, and 7.5%). The values of MDD increased with increasing Halloysite nanotubes percentages, while the optimum moisture content reduced as these percentages increased. The drop in OWC is due to Halloysite's propensity to fill pores between gypseous soil particles, occupying the gaps initially filled by water in the untreated gypseous soil. The particle size, changes in sample surface area, and specific gravity of the Halloysite and original soil lead to a rise in the MDD.

Table 3: Compaction test results for Halloysite nanotubes with soil.

Halloysite Nanotubes Ratio, %	MDD, gm/cm^3	OWC, %
0	1.810	12.60
2.5	1.827	12.31
5	1.840	11.95
7.5	1.891	11.70

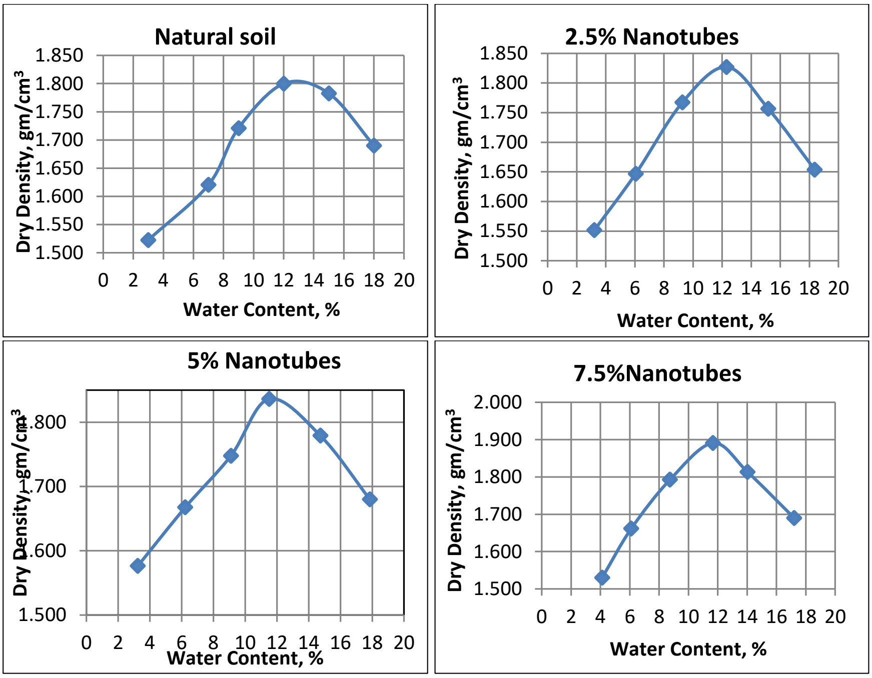

Figure 2: The compaction test for untreated and treated gypseous soil with (2.5, 5, 7.5%) Halloysite.

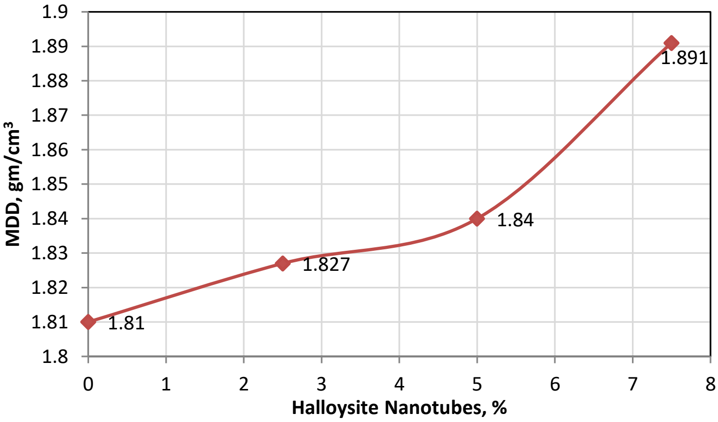

Figure 3: The relationship between maximum dry density and Halloysite Nanotubes (0, 2.5, 5, and 7.5) %.

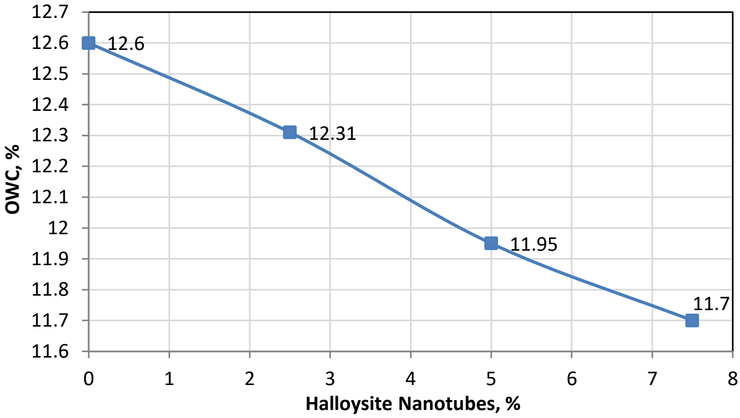

Figure 4: The relationship between optimum water content and Halloysite nanotubes (0, 2.5, 5, and 7.5) %.

4.2 The Effect of Halloysite Nanotubes on Shear Strength

The direct shear test results are represented by the relation between shear displacement and shear stress shown in Figure 5**.** Also, the relation between normal stress (σ) (50,100, 200) kPa and maximum shear stress (τ_{max}) is shown in Figure 6. This figure illustrates that the cohesion increase with increasing the Halloysite content. At the same time, the friction angle decreases with Halloysite content with a maximum value of 7.5% nanotubes and minimum value of 2.5% nanotubes because the particle density of Halloysite nanotubes is high. The effective contact area between the soil particles and the Halloysite or the effective contact area between the soil particles plays an important role in the interaction mechanism between soil and Halloysite. The effective contact area is likely related to the particle size of the soil, the shape and size of Halloysite openings, and the dimension of the Halloysite. On the other hand, as shown in Table 4, the friction angle increases with Halloysite concentration at 5% nanotubes because Halloysite reduced porosity in the gypseous matrix by refilling spaces between the gypseous particles, thus attaching the gypseous particles for the treated soil.

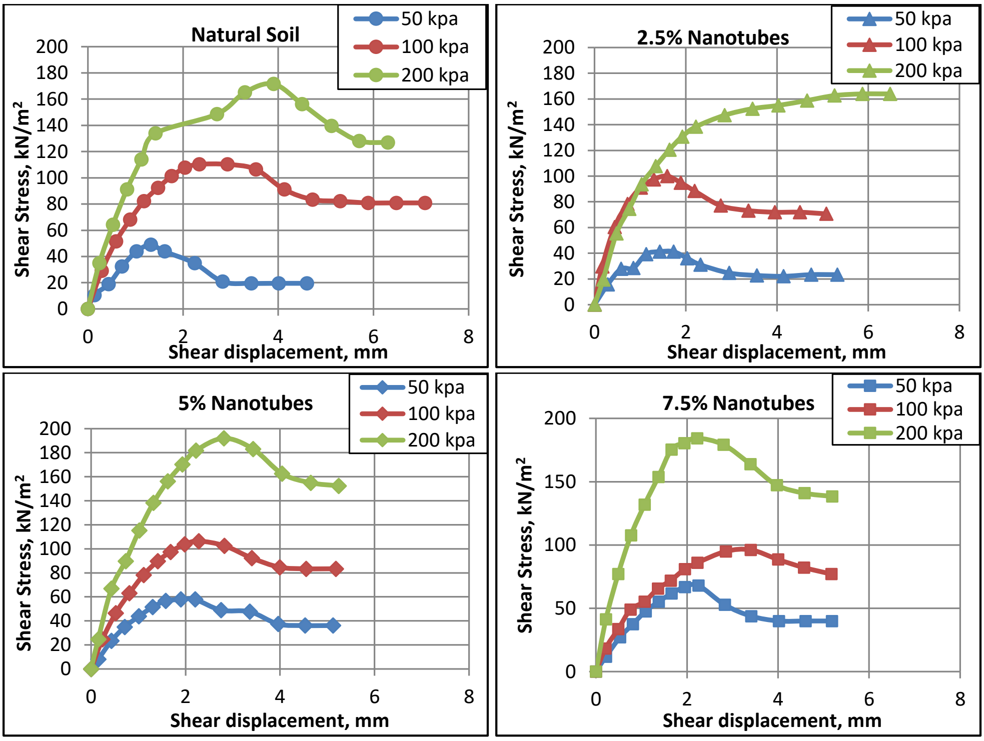

Figure 5: Shear displacement and shear stress relationships for untreated and treated gypseous soil with (2.5, 5, 7.5) % Halloysite Nanotubes.

Table 4: Shear parameters of treated and untreated soil obtained from direct shear tests.

Halloysite nanotubes ratio, %	Cohesion, C (kN/m^2)	Friction angle, ϕ (°)
0	7.2	40.81
2.5	9.5	38.51
5	15	41.62
7.5	25	38.05

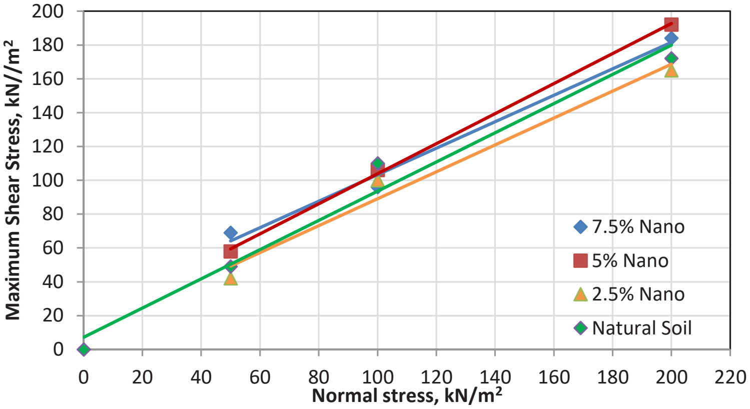

Figure 6: Normal stress versus maximum shear stress for untreated and treated gypseous soil with (0, 2.5, 5, 7.5) % Halloysite nanotubes.

4.3 The Effect of Halloysite Nanotubes on Collapsible Test

The collapse test results suggest that the soil's CP is 3.42%, classifying it as moderately troubled according to [21]. Figure 7 illustrates the results of a single collapse test on samples of soil that have been saturated in gypsum and treated with nanotubes at a 2.5% concentration under 200 kPa of pressure. It can be seen that the collapse potential dropped to 0.53% while the vertical strain increased from 2.84 to 3.37%. Figure 8, which shows the results of a single collapse test on gypsum soil samples treated with 5% nanotubes, reveals that the vertical strain changed from a value of 2.26% to 2.95% at 200 kPa, making the CP 0.69. Figure 9 shows the results of a single collapse test on gypsum soil samples that were treated with 7.5% nanotubes. Due to the vertical strain changing from 3.63% to 4.68% due to adding 200 kPa (stress), the CP was reduced to 1.05%. Halloysite form another sort of bond that resists and reduces gypseous soil collapsibility by surrounding the gypseous soil particles and filling the voids, preventing water from reaching them, thus reducing the solubility of gypsum and making soil particles more cohesive. The chemicals found in Halloysite can form strong reinforcing bonds with soil. Therefore, the collapse potential of soil was decreased.

Table 5: Findings from a single collapse test.

Halloysite nanotubes ratio %	CP (%)	Change in CP (%)
0	3.42	
2.5	0.53	84.5
5	0.69	79.82
7.5	1.05	69.30

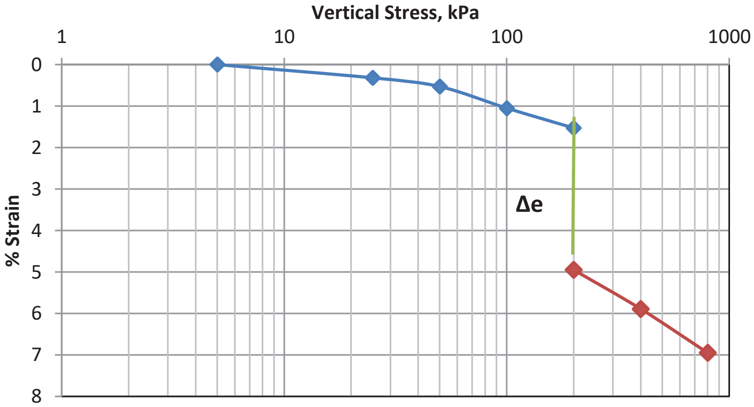

Figure 7: Findings of the untreated soil collapse test.

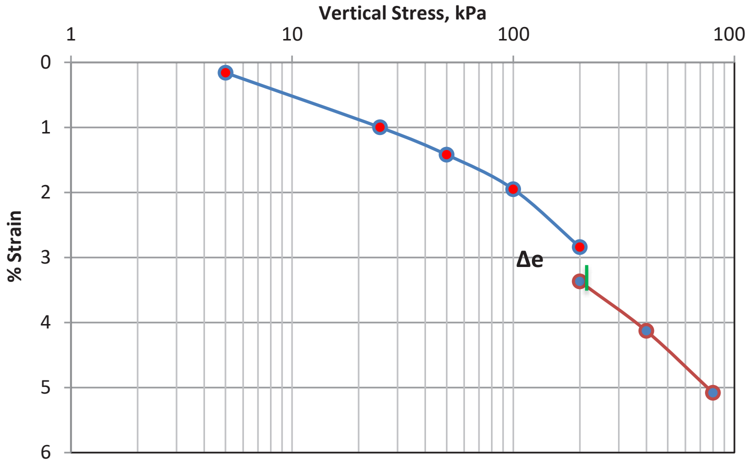

Figure 8: Results of the collapse test on the 2.5% nanotube-treated gypseous soil.

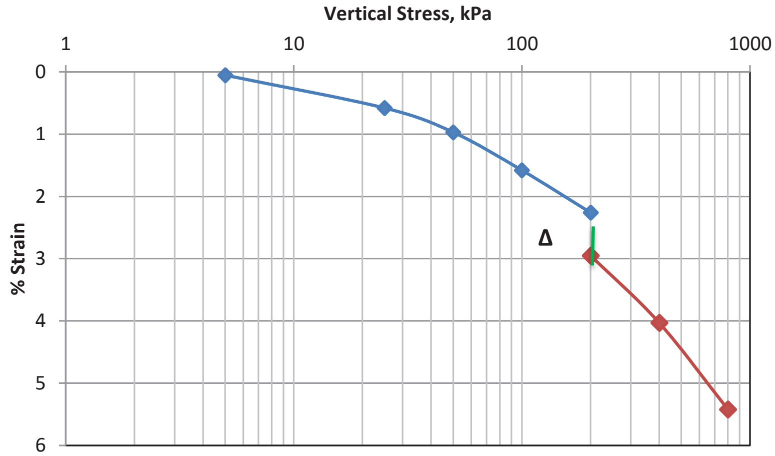

Figure 9: Results of the collapse test on the 5% nanotube-treated soil.

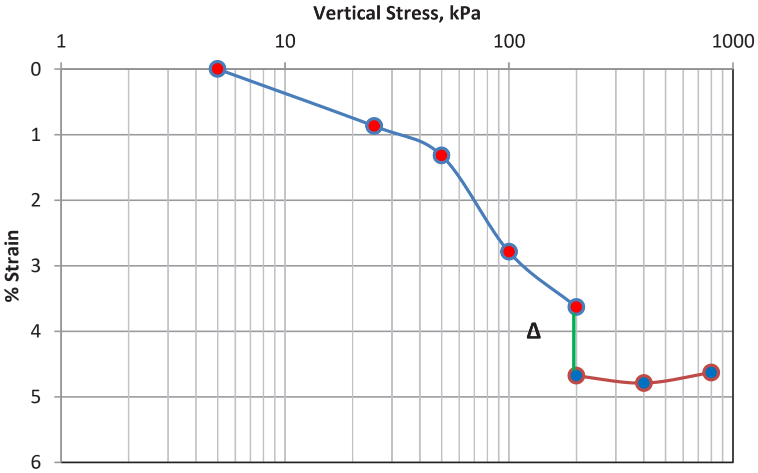

Figure 10: Results of the collapse test on the 7.5% nanotube-treated gypseous soil.

4.4 The Effect of Halloysite Nanotubes on CBR

The most used test for determining the bearing capability of subgrade soils is the California bearing ratio (CBR) test. For unsoaked and soaked circumstances, it measures the force required to cause a plunger to penetrate (2.5 or 5 mm). In this section, the findings from these tests are presented and discussed.

4.5 A CBR for Soil, Halloysite, and Their Mixing (Unsoaked Condition)

The main objective of the test is to investigate the actual CBR values for the treated and untreated gypseous subgrade layers. The effect of adding Halloysite in different proportions (0, 2.5, 5, and 7.5) % with soil on unsoaked California bearing ratio (CBR) is shown in Figure 11. As the CBR values of untreated and treated soil increase with increasing penetration, the CBR value for untreated at penetration of 2.5 mm is 2.93% and increases to 9.85% at penetration of 5mm. For soil with 2.5% of Halloysite, the CBR at 2.5 mm is 13.71%. This value increases to 31.46% at a penetration of 5 mm. For soil with 5% of Halloysite, the CBR at 2.5 mm is 11.27%. This value increases to 23.76% at a penetration of 5 mm. It observed decreases in the CBR value treated with 7.5% of Halloysite compared with other portions. The CBR strength could have decreased at 7.5% Halloysite because of agglomeration and defects caused by the dispersion of the nanotubes. In nanomaterials, the surface area to volume ratio is high, thus, producing high levels of adhesion forces between neighboring nanoparticles as well as high levels of particle interaction energy. Agglomeration results from this phenomenon. The nanomaterials agglomeration reduces soil resistivity. Table 6 shows these results.

Table 6: Unsoaked CBR test results.

Halloysite Ratio, %	Unsoaked CBR
0	9.85
2.5	31.46
5	23.76
7.5	11.96

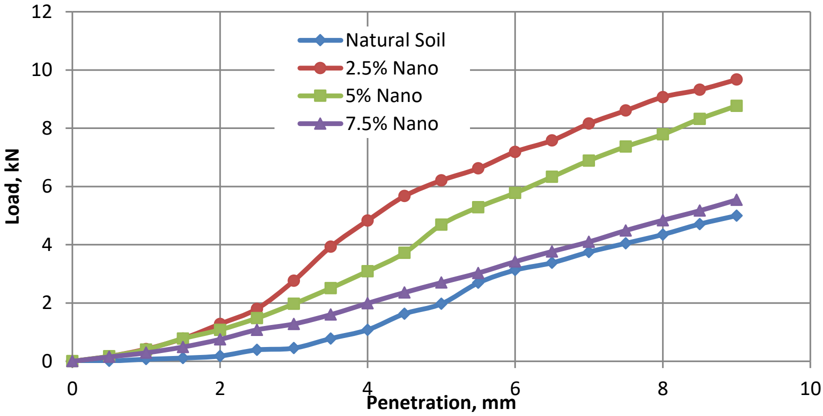

Figure 11: Unsoaked CBR test results for untreated and treated gypseous soil with (2.5, 5, and 7.5) % of Halloysite.

4.6 CBR for Soil, Halloysite, and Their Mixing (Soaked Condition)

The effect of adding Halloysite with soil on the soaked ratio is shown in Figure 12. It can be seen that the CBR values of untreated and treated soil increase with increasing penetration. The CBR value for untreated at the penetration of 2.5 mm is 1.65%, increasing to 6.08% at the penetration of 5 mm. The effect of 2.5% of Halloysite with soil, the CBR at 2.5mm is 12.19%. This value increases to 24.37% at a penetration of 5mm. For soil with 5% of Halloysite, the CBR at 2.5mm is 11.13%; this value increases to 20.16% at a penetration of 5 mm. While seen decreases in the CBR value treated with 7.5% of Halloysite compared with other portions due to gather and agglomeration of nanotubes together when the amount of nanoparticles is increased because of their high surface area. In this case, they do not work on the bonding and cohesion of the soil particles. Therefore, when the soil is soaked in water, the bond between its particles is lost, so the gypsum dissolves, and the CBR percentage decreases. Table 7 shows these results.

Table 7: Soaked CBR test results.

Halloysite Ratio, %	Soaked CBR, %
0	6.08
2.5	24.37
5	20.16
7.5	5.37

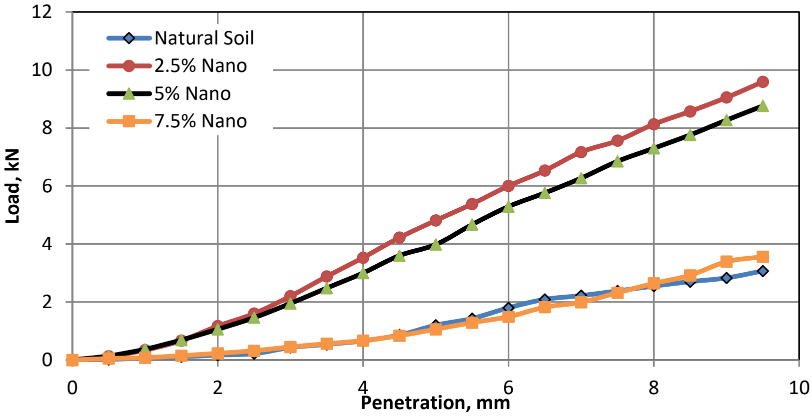

Figure 12: Soaked CBR test results for untreated and treated gypseous soil with (2.5, 5, and 7.5) % of Halloysite.

5. CONCLUSIONS

Based on the results presented in this study which was conducted on the soil samples taken from Ayn al-Tamr in Karbala province, the following conclusions can be drawn:

- The maximum dry density increases with the increase in the content of Halloysite nanotubes which is equal to (1.810, 1.827, 1.840, and1.891) gm/cm^3 at (0, 2.5, 5, and7.5) % of Halloysite, respectively. At the same time, the optimum moisture content decreases with increasing the content of Halloysite nanotubes which equals (12.60,12.31,11.95, and 11.70) % at (0, 2.5, 5, and7.5) % of Halloysite, respectively.
- The higher the content of Halloysite, the greater the soil cohesion equal to (7.2,9.5,15 and 25) kPa at (0,2.5,5 and7.5) % of Halloysite content, while the angle of shearing resistance increased by 41.62 kPa at 5% Halloysite content and decreases (38.51 and 38.05) kPa at (2.5 and 7.5) % of halloysite content, respectively.
- The value of collapse potential (C.P %) decreases from 3.42% to (0.53,0.69 and 1.05) % at (2.5, 5, and 7.5) % of the Halloysite content increases. The collapse potential shifts from moderate trouble to no problem when the Halloysite increases up to (7.5%). The optimum content of Halloysite that was observed during the test is 2.5%.
- The maximum value of unsoaked and soaked (CBR) are (31.46 and 24.36) %, respectively, obtained at (2.5%) of Halloysite content. The CBR increased with the Halloysite content increase until the (5%) Halloysite content is equal (23.76 and 20.16) %for soaked and unsoaked, respectively. While CBR decreases with Halloysite content increase (7.5%) equals (11.96 and 5.37) % for soaked and unsoaked, respectively.

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