

Leaching-Permeability Behavior of Collapsible Gypseous Soils Treated with Nano-Titanium Dioxide

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Abstract

As a result of the limited studies that have been conducted on the utilization of nano titanium dioxide as a nanomaterial for stabilizing gypseous soils in geotechnical works, this study is directed to predict the changes in the coefficient of permeability k , the leaching strain, the total dissolved salts TDS, and the pH values with the changes in the percentages of nano titanium dioxide NTD. The gypseous soil samples were obtained from three sites located north of Baghdad, the capital of Iraq, with different gypsum contents of about 34%, 50%, and 60%. Tests have identified the mechanical and physical characteristics of the studied gypseous soils. In addition, oedometer permeability leaching tests were conducted using an oedometer cell apparatus. The results of the tested gypseous soils presented a significant effect of NTD on reducing the coefficient of permeability k and cost-effectively, especially at 0.3 and 0.5% for the three tested soils. For S1 tested soil, the reduction percentages of the k values were 79.02% and 80.0% when treated with 0.3% and 0.5% of NTD, respectively. While for S2 tested gypseous soil, the reduction percentages were 75.9% and 79.1%, and 66.04% and 73.6% for S3 tested gypseous soil when treated with 0.3% and 0.5% of NTD, respectively. The treated gypseous soils are exposed to less gypsum dissolution, as the NTD material forms an impermeable layer to prevent direct contact between water and gypsum. This reduces gypsum dissolution and, thus, reduces leaching strain. For S1 tested soil, the percentage of reduction of the leaching strain was 90.5%, while for S2 and S3 tested soils, it was 91.2% and 89.9%, respectively, when 0.3% of NTD was applied. As the percentage of the NTD increased for S1, S2, and S3, the pH values decreased due to decreased TDS in the leached water, and it is clear that 0.3% of NTD gives a reliable pH value for the three tested soils. Considering these results, it appears that even small amounts of nano titanium dioxide have the potential to be an effective agent for reducing permeability and stabilizing collapsible gypseous soils in civil engineering projects, compared with other nano or traditional materials.

Keywords: Nano Titanium Dioxide; Gypseous Soils; Coefficient of Permeability; Stabilization; FE-SEM.

1. Introduction

In most regions of the world, natural soils and aggregates contain varying quantities of soluble salts [1, 2]. Gypsum is one of these soluble salts, which has a detrimental effect on pavement and earth structures. Gypsum and Sodium Chloride represent the very common salts in Iraq [3-6]. Gypsum is a hydrated calcium sulphate $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ found in many other forms, Bassanite $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$ and Anhydrite CaSO_4 [7]. Hesse [8] and Kuttah & Sato [9] stated that pure gypsum contains 20.9% of combined water H_2O , 46.6% sulphur trioxide SO_3 , and 32.5% calcium oxide CaO . Gypsum has a low specific gravity of 2.32. This relatively low value dramatically influences the physical and mechanical

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properties of soils containing large amounts of gypsum material [10, 11]. However, an increase in the groundwater table, leaks through liners or pipelines, infiltration of water from rainfall, and other factors can cause gypseous soils to dissolve and soften. Because the elements of the structure cannot follow the abrupt deformation caused by the rearrangement of internal forces or stresses, this can result in severe damage or even the collapse of buildings erected on or in such soils [12, 13]. Because gypsum provides a joining effect, gypseous soils are typically stiff when dry. Because of their solubility properties, they significantly decrease in strength and increase in compressibility when they come into contact with water [14-16].

Due to gypsum's ability to give apparent cementation when the soil is dry, gypseous soils are typically categorized as collapsing soils. However, the dissolving and softening brought on by the presence of water usually result in severe structural failure. Any unsaturated soil that, when wet, has a dramatic reorganization of particles and a notable reduction in volume is considered collapsible soil. The amount of collapse and its rate depend on the materials' mineralogy, the shape and grain size distribution of bulky grains, natural moisture content, void ratio, and a cementing agent. Some soils collapse upon soaking without an additional load, while others require an additional surcharge load for collapse to occur [17-21].

Significant strength losses upon wetting, an abrupt increase in compressibility upon wetting, ongoing deformation and collapse upon leaching due to water movement, the presence of cracks resulting from seasonal variations, and the existence of sinkholes in the soil as a result of the local dissolution of salts like gypsum are the problems associated with Iraqi soils that contain large amounts of gypsum, roughly 31% [10, 22, 23]. The problem becomes more severe when the water flows inside the soil, resulting in soil mass loss due to the leaching of gypsum. Leaching is when fluids' natural or artificial penetration into soil results in the solution and washing of soluble soil components [24]. Leaching is the process where water flows through the soil. Also, leaching is defined as the solubility of the gypsum in the soil by the water table or water flow into the soil [25]. Namiq & Nashat [26] reported that the strain was reduced by increasing the stress on the soil during the leaching, and the permeability of the soil decreased due to the new arrangement of soil particles over leaching time. Ahmad et al. [27] stated that leaching occurs when water flows through soil to remove soluble minerals and salts. After leaching, soluble material-containing soils continuously change their engineering characteristics. Buildings built on or inside these soils face serious challenges due to these changes.

The leaching decreases the contact area between the particles and breaks the cementitious bonds of the gypsum salts, so the voids increase [11, 28]. In soaking cases of short-term flooding, gypseous soils are compressible and sufficiently reliable soil base, while in the case of long-term flooding, settlement develops due to the dissolution of salts and gypsum. The settlement's magnitude and rate depend on the initial gypsum content, relative amounts of leached salts, the mineralogy and type of soil and soil properties, and acting load [27, 29, 30]. Additionally, gypsum leaching can result in an extensive amount of progressive and unrecoverable compressive strain even when the soil has as little as 5% gypsum content. The leaching strain is connected with gypsum dissolving and removal, and the collapse of the soil fabric. The gypseous specimens' leaching strain was two to five times higher than the soaking strain [31].

Nanotechnology is an innovative method that describes nanomaterials in addition to weak natural soil for filling the nano-level voids and improving geotechnical properties [32]. Even though various conventional ground improvement techniques are available, the nanomaterial-treated soils proved to make the treatment cheaper due to the addition of a very low dosage of nanomaterial.

Several earlier studies examined the impact of various percentages of nanomaterials, such as nano clay and nano silica, on soil geotechnical characteristics, focusing on soil strength, swelling and permeability. Majeed et al. [33] conducted a study to investigate the effect of applying three nanomaterials, nano MgO, nano CuO, and nano Al₂O₃, on some of the soft soil geotechnical characteristics. The tested geotechnical characteristics included water content, dry density, and unconfined compressive strength. The results indicated that the increment in the nanomaterials content improved both the compressive strength and the dry density, while lowering the soil mixtures' moisture content. Vijayan & Jose [34] stated that unconfined compressive strength (UCC) increases with the rise in the percentage of Nano MgO by up to 1% and decreases with further addition.

Albusoda & Khdeir [35] stated that the improvement results showed that the optimum percentages of nano fly ash and silica fume for the collapse potential reduction of natural gypseous soil with a gypsum content of 58% were 2% and 4%, respectively. The collapse potential decreased from 13.6% to 2.16% with an increase in the silica fume percentage to 4% by soil dry weight, and decreased to 1.29% with an increase in the nano fly ash percentage to 2%.

Hayal et al. [36] demonstrated that when adding nano-silica to gypseous soil with 47% gypsum content, the collapse potential decreased whenever the nano-silica increased until 1%, at which point a further stabilizer increased the collapse potential. The percentage reduction in the collapse potential is about 91%, and the effect of adding nano-silica changed the classification of the severity of collapse from moderate trouble to no problem.

The study of Nano Titanium Dioxide TiO₂ as a new soil amendment material is a hot research direction that has attracted many researchers from the international geotechnical engineering circle [37-39]. Nano titanium dioxide creates nontoxic and non-environmental pollution, and the cost of production is relatively cheap, making large-scale manufacturing available. It is expected to be a promising alternative to traditional additives [40, 41]. Nano titanium

dioxide is used as an additive where the soil faces the problem of low shear strength, high collapsibility and compressibility, which is unfavorable from a geotechnical point of view [42, 43]. Jili et al. [40] showed that nano TiO_2 can substantially raise the liquid and plastic limits of TiO_2 -treated clayey silt but diminish its plasticity index to some degree. Compared with untreated samples, the standard proctor compaction test results show that the optimum water content increases and the maximum dry bulk density decreases. The extremely small particle size of nano TiO_2 may be the primary source of its impact on the physical performance of clayey soil.

Due to a critical gap remains uninvestigated and a lack of research in utilizing nano titanium dioxide as a nanomaterial physical and chemical stabilizer for gypseous sandy soils in geotechnical works (permeability leaching test), this study aims to forecast how variations in the percentages of nano titanium dioxide will affect the coefficient of permeability k , the leaching strain, the total dissolved salts TDS, and the pH values. A scanning electronic microscope FE-SEM was used to investigate the micro changes in the tested soil particle structure before and after leaching. Three types of gypseous soils from different parts of Iraq were experimented with in this study. The oedometer cell apparatus was used to perform oedometer permeability leaching tests on samples already treated with nano titanium dioxide at different percentages.

This article begins with an introduction that outlines the study's context and significance. The methodology section follows, detailing the experimental procedures and materials used. The results section presents the main findings, which are then discussed in relation to existing literature. Finally, the conclusion summarizes the main outcomes.

2. Experimental Works

2.1. Methodology

Physical, chemical, and mechanical analysis were carried out to examine the performance of three collapsible gypseous soils and assess the impact of adding nano titanium dioxide, as shown in Figure 1. For each soil type and test, a pair of soil samples was meticulously prepared under identical conditions and additive percentages to obtain average values of the test results and enhance accuracy.

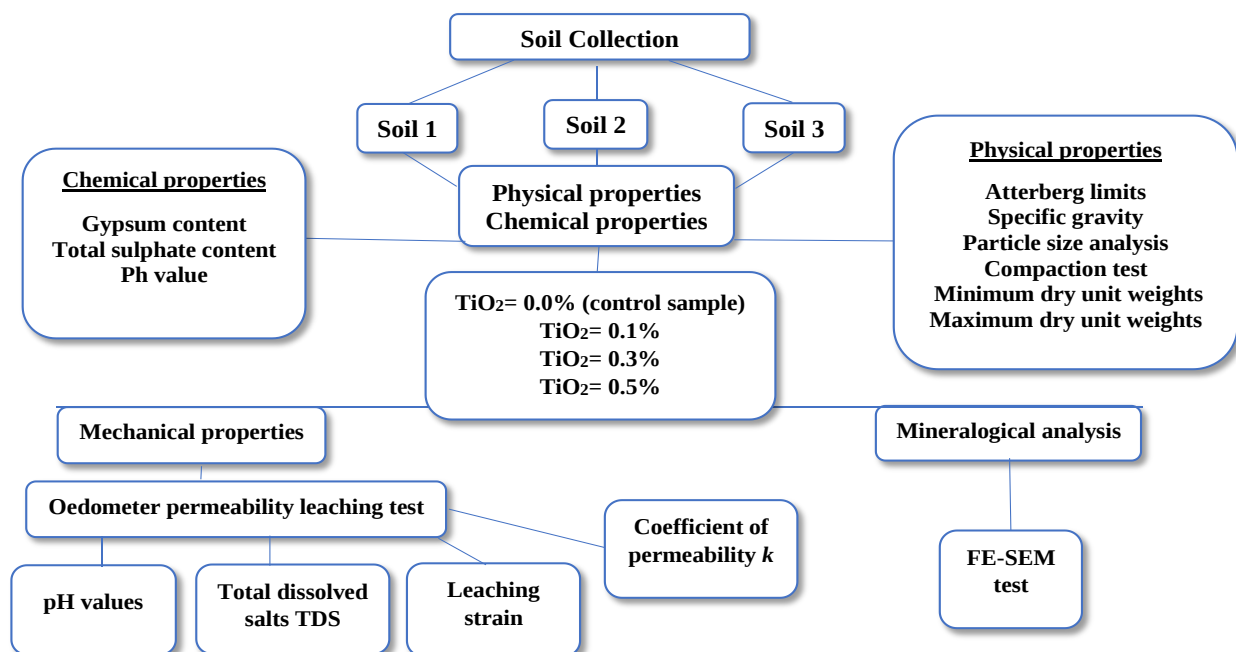


Figure 1. Methodology flow chart

2.1.1. Physical and Chemical Tests

The physical and classification tests of the selected gypseous soils were conducted following the standards to determine the fundamental soil indices like moisture content, Atterberg Limits (liquid limit, plastic limit, and hence plasticity index), specific gravity, and sieve analysis. Because the soils include a percentage of gypsum content, kerosene has been used instead of distilled water in the specific gravity tests to prevent gypsum dissolution during the test [44-46, 16]. A scanning electronic microscope FE-SEM was used to investigate the micro changes in the tested soil particle structure with nano titanium dioxide before and after soaking.

The chemical properties of the tested gypseous soils were evaluated through a standard series of tests, and two methods were used to measure the gypsum contents in the soils: The approximate method and the Al-Muftly & Nashat Method [22].

2.1.2. Oedometer Permeability Leaching Test

This study focuses on leaching untreated tested gypseous soils S1, S2, and S3, as well as treated soil samples containing varying percentages of nano titanium dioxide NTD 0.1, 0.3, and 0.5% at a given relative density and natural water content. The leaching process is carried out in an oedometer cell at a pressure of 200 kPa, as shown in Figure 2. The samples were loaded gradually with time until they reached the vertical stress of 200 kPa, then saturated from the bottom and left for 24 hours. After that, the leaching continued for seven days, allowing the water to enter from the bottom and the outlet of the water from the top. The coefficient of permeability k was measured with time, as well as the leaching strain, the total dissolved salts TDS, and the pH value of the leached water that accumulated in the cylinder. The TDS and pH values can be measured by inserting the specified device into the beaker of the leached water, as illustrated in Figure 3.



Figure 2. Oedometer permeability leaching test samples

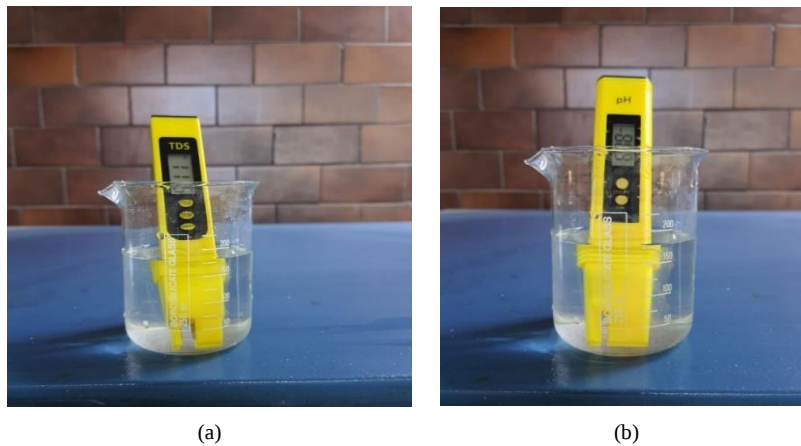


Figure 3. (a) TDS device; (b) pH device

2.2. Materials

2.2.1. Soils

The natural gypseous soils used in this research were collected from three different locations in Tikrit City, which is located 180 kilometers north of Baghdad City, Iraq's capital city. These soils contained varying amounts of gypsum. The samples were collected from 1.5 to 2.5 meters beneath the ground surface. An excavator collected disturbed soil samples and stored them in durable nylon bags until the start of the testing procedures. The physical and classification tests of the selected gypseous soils, designated S1, S2, and S3, followed the specifications for determining the essential soil indices measured. Table 1 summarizes the particle size analysis of the tested gypseous soils following the ASTM D422-63(2007) [47], and ASTM D2487-11 [48] specifications. Following the unified soil classification system USCS, sands with 5–12% fines require dual symbols; therefore, S1 is classified as poorly graded sand with silt and gravel SP-SM, and S2 is classified as well-graded sand with silt and gravel SW-SM. In contrast, S3 with fines less than 5% is classified as poorly graded sand with gravel SP gypseous soils. The uniformity coefficients C_u are 5.0, 6.67 and 5.8, while the curvature coefficients C_c are 0.8, 1.1 and 0.93 for S1, S2 and S3, respectively.

Table 1. Particle size analysis of the tested gypseous soils

Tested Soils	Soil 1	Soil 2	Soil 3
Gravel %	22.0	17.6	24.4
Sand %	70.8	72.4	71.0
Fines %	7.2	10.0	4.6
C _u	5.0	6.67	5.80
C _c	0.8	1.1	0.93
Classification (USCS)*	SP - SM	SW - SM	SP

* USCS: Unified Soil Classification System

The Atterberg limits, which include the plasticity index, liquid limit, and plastic limit, are presented in Table 2 and are obtained following ASTM D4318-17e1 [49]. From the particle size analysis, the three gypseous soils exhibited no plastic limits. The specific gravity values of the examined gypseous soils were measured by ASTM D5550-14 [50]. The findings indicate that the specific gravity values for the untreated tested gypseous soils S1, S2, and S3 are 2.62, 2.59, and 2.55, respectively. Specific gravity tests indicate a reduction in specific gravity values of the examined gypseous soils as gypsum content increases. The specific gravity values for the three studied gypseous soils fall below the standard limits of 2.65-2.67 [51], especially in S2 and S3 with higher gypsum contents due to the influence of gypsum's low specific gravity of 2.32 [10, 11]. A low natural moisture content was found in the examined soils [52], which may reach a dry state. This is regarding the soil sampling season in a hot, dry climate that is far away from the groundwater table levels.

Table 2. Physical properties of the tested gypseous soils

Tested Soils	Soil 1	Soil 2	Soil 3
Specific gravity G _s	2.62	2.59	2.55
Liquid limit %	21	23	20
Plastic limit %	NA	NA	NA
Plasticity index %	21	23	20
Natural Water Content W %	1.3	1.8	2.0
Collapse potential value C _p	7.77	8.45	12.24

Table 3 establishes the chemical characteristics of the studied gypseous soils. Variations in gypsum content were noted because the soil samples were collected from three distinct locations. The studied gypseous soil S1 had the lowest gypsum level at approximately 34%, whereas S2 and S3 exhibited greater gypsum amounts of around 50% and 60%, respectively.

Table 3. Chemical characteristics of the tested gypseous soils

Chemical properties	Soil 1	Soil 2	Soil 3	Specifications
pH value	8.10	8.17	8.21	BS 1377: 1990 [53]
Total sulphate content SO ₃ %	15.90	23.49	27.99	BS 1377: 1990 [53]
Gypsum content %	34.19	50.51	60.17	Approximate method
	34.82	49.98	59.63	Al-Muftly & Nashat [22] method

2.2.2. Nano titanium dioxide NTD

Experiments were carried out to examine the impact of employing nanomaterials in stabilizing gypseous soils. The primary characteristics of the nano titanium dioxide utilized in this study are displayed in Table 4, and it was supplied by the US Advanced Nano Material Provider Company, Houston, US Research Nanomaterials. The nano titanium dioxide NTD is a rutile type, white powder with a low density of about 4.25 g/cm³. The findings of the study's FE-SEM for NTD are shown in Figure 4. A nearly spherical structure with an average particle size diameter of 37 – 44 nm is seen in the FE-SEM image of NTD.

Table 4. Nano Titanium Dioxide Properties

Property	Appearance	Formula	Purity	Average particle size	Type	Density (g/cm ³)
Value	white	TiO ₂	99 %	30 - 50 nm	Rutile	4.250

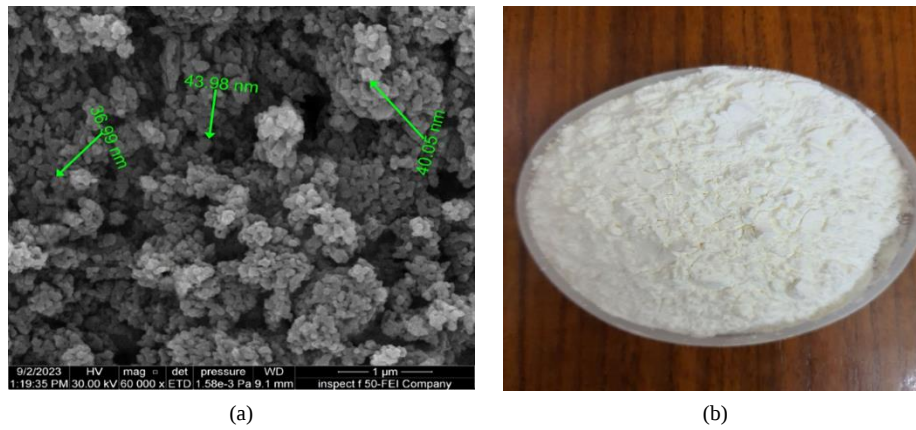


Figure 4. (a) FE-SEM result for the Nano Titanium Dioxide (b) Nano Titanium Dioxide

3. Experimental Results and Discussion

Figures 5 to 7, respectively, illustrate the change in the coefficient of permeability k with time for untreated and treated gypseous soil samples S1, S2, and S3 with 0.1, 0.3, and 0.5% of NTD.

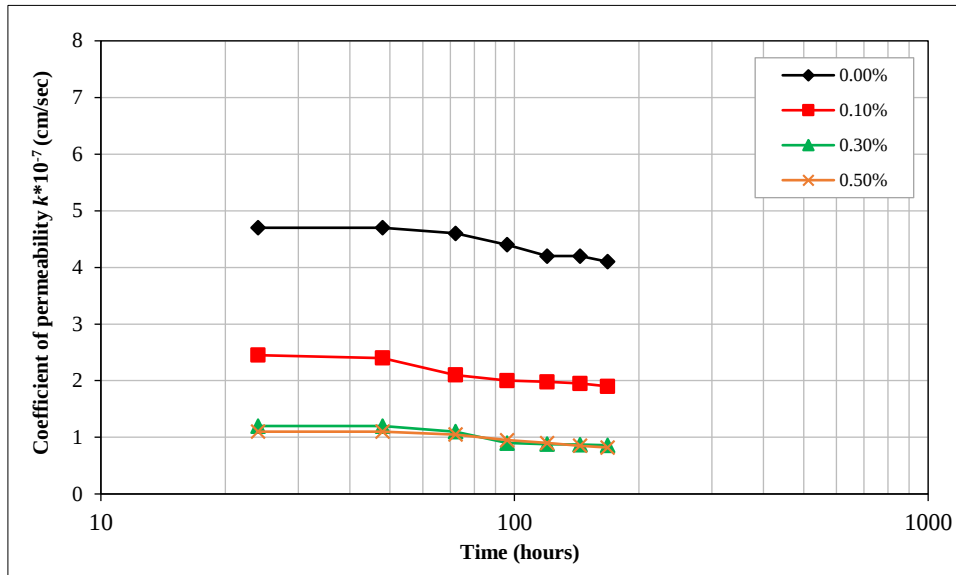


Figure 5. The variation of the permeability coefficient with time for S1 soil

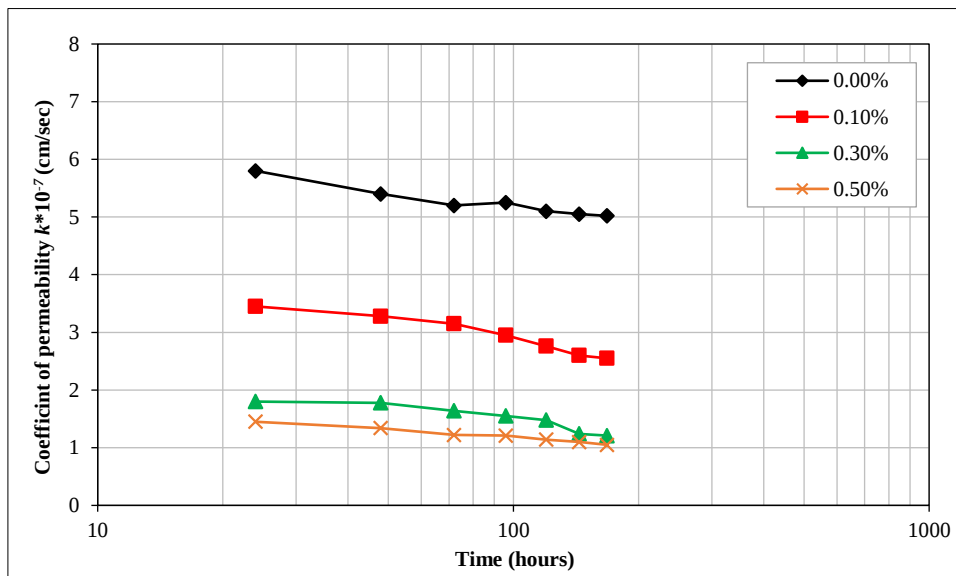


Figure 6. The variation of the permeability coefficient with time for S2 soil

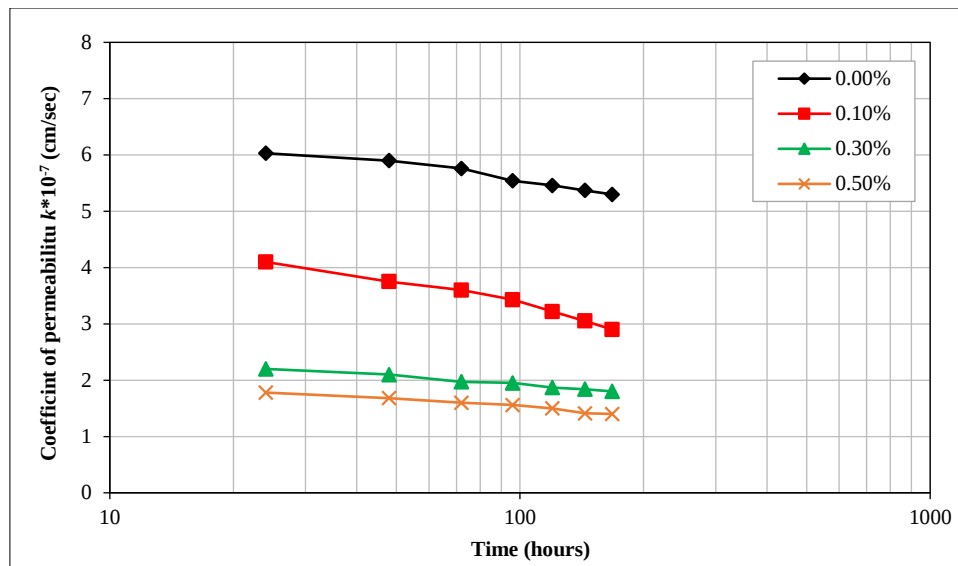


Figure 7. The variation of the permeability coefficient with time for S3 soil

Gypsum functions as an effective cementing agent in gypseous soil samples, mainly when the environment is dry and there is a higher gypsum content. Upon analyzing the oedometer permeability leaching test findings after 24 hours, it is evident that the leaching process in untreated gypseous soils, at varying gypsum concentrations, results in the degradation of some or all of the cementing bonds that come into contact with the soil particles. Furthermore, the dissolution of gypsum results in an increase in the voids ratio, hence the coefficient of permeability k , as shown in Figures 8 to 10. Figure 11 illustrates that when the test first started, for untreated gypseous soils S1, S2, and S3, the big voids grew in parallel with the rise in gypsum content. The coefficient of permeability k value for the untreated S3 soil at the beginning of the test is the highest, 6.03×10^{-7} (cm/sec), compared with the coefficient of permeability values for the other two gypseous soils S1 and S2, 4.7×10^{-7} (cm/sec) and 5.8×10^{-7} (cm/sec) respectively. This is explained by the fact that in relation to S1 and S2, the dissolved gypsum content in gypseous soil S3 reaches its maximum value. After that, as the leaching process proceeds, the void ratios for each investigated gypseous soil decrease by a different amount.

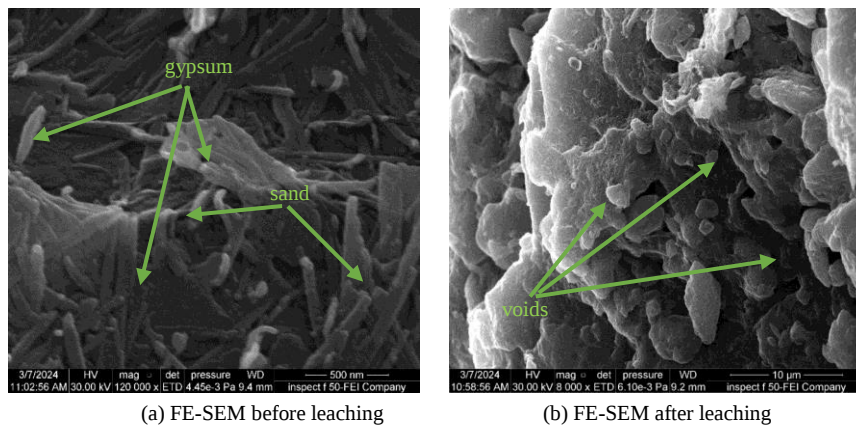


Figure 8. FE-SEM for untreated gypseous S1 soil before and after leaching

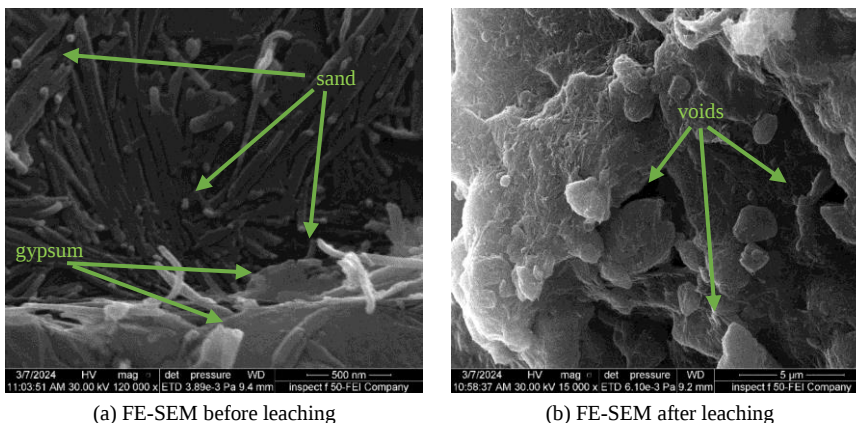


Figure 9. FE-SEM for untreated gypseous S2 soil before and after leaching

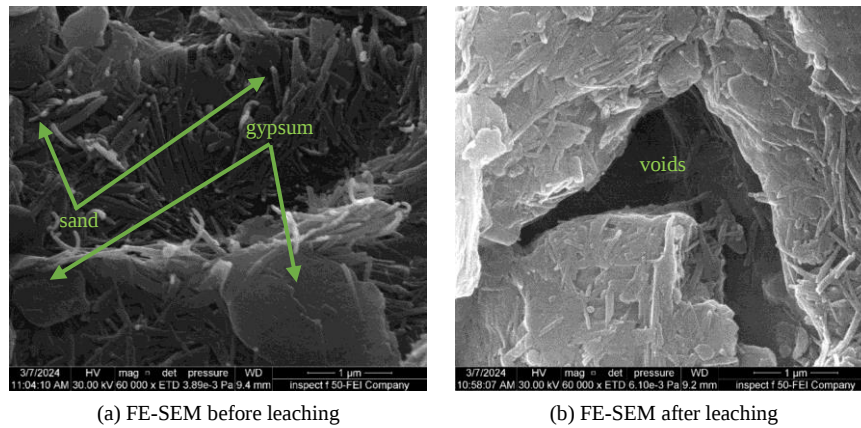


Figure 10. FE-SEM for untreated gypseous S3 soil before and after leaching

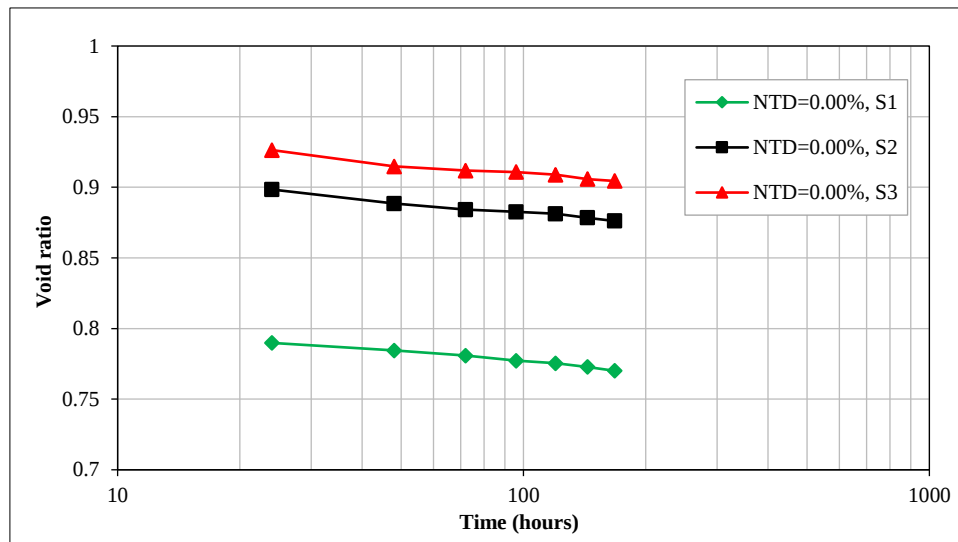


Figure 11. The variation of the void ratio for untreated gypseous soils versus the leaching time

Similar findings were made by Fattah et al. [54] and Al-Riahi et al. [55], who recommended that the leaching process's corresponding modifications to the studied gypseous soils' structures led to the formation of enormous voids, which grew in size as the gypsum concentration increased. With an increase in the test time and the dissolution of gypsum particles during the leaching process, the permeability coefficient gradually declined. This behavior could be explained by the dissolution of gypsum particles, causing the cavities to crumble and pore channels to get blocked, resulting in a collapsible structure. Figure 12 supports the conclusions of Snodi [56], Al-Gharbawi [57] and Fattah et al. [54] that a greater collapse potential value corresponds to a more significant coefficient of permeability.

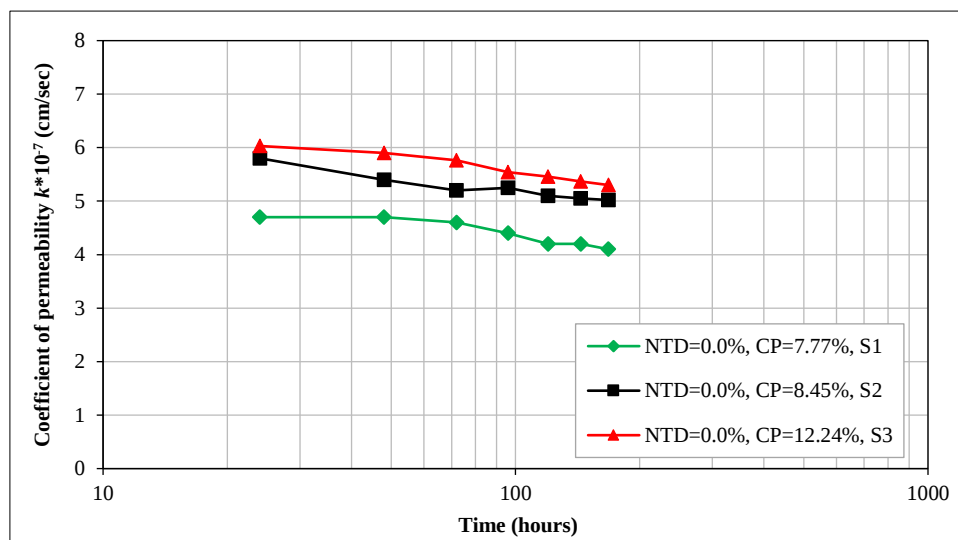


Figure 12. The fluctuation of the coefficient of permeability with time for untreated tested gypseous soils

The findings demonstrate that adding nano titanium dioxide to the studied soil samples at all percentages 0.1, 0.3, and 0.5% significantly decreases the coefficient of permeability k at different rates in the gypseous soil samples. This might be explained by the way that NTD particles' nano size decreases the tested soils' porosity, eliminates water pathways and forms an impermeable layer, which in turn reduces the permeability of the improved gypseous soils, as shown in Figures 13 to 15. The impermeable layer formed by nano titanium dioxide in the tested gypseous sandy soil results from both physical clogging (pore filling) and chemical modification by bonding. Both mechanisms work together to reduce water permeability and improve soil stability significantly. Pore Filling results from the fact that NTD particles are extremely small and capable of filling the voids between sand grains, physically blocking the pathways of water that normally would be used to move through the soil. This reduces the soil's water absorbency and permeability. Moreover, NTD has surface hydroxyl groups (-OH) on the surface (called Ti-OH groups). These groups make NTD chemically active, meaning they can interact with other ions or particles in the soil and the reactions happen depending on the pH of the soil acidic or alkaline. NTD can bond to the surfaces of the soil particles or gypsum crystals through hydrogen bonding or ion bridging. This creates particle to particle connections that strengthen the soil structure.

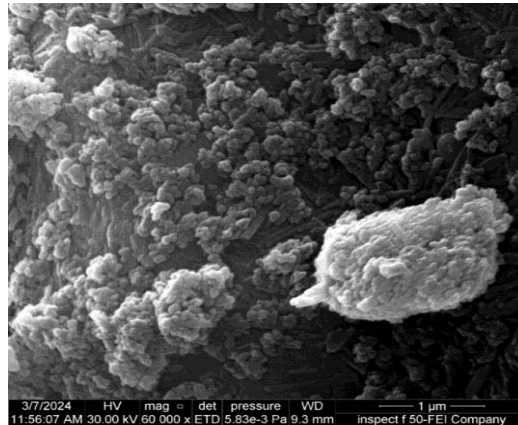


Figure 13. FE-SEM for treated gypseous S1 soil by 0.3% of NTD

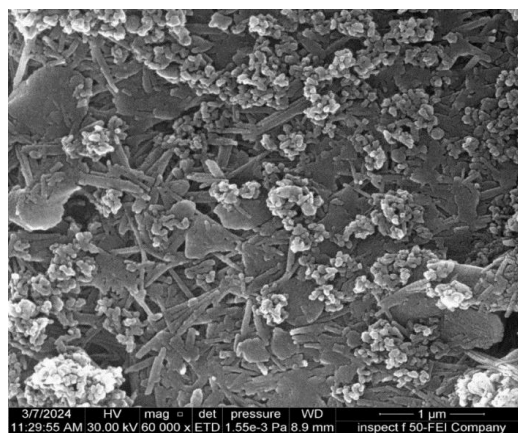


Figure 14. FE-SEM for treated gypseous S2 soil by 0.3% of NTD

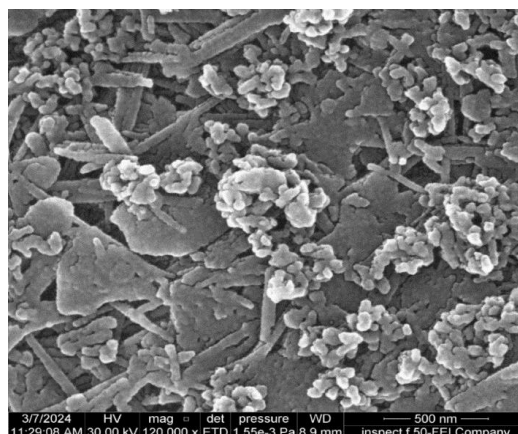


Figure 15. FE-SEM for treated gypseous S3 soil by 0.3% of NTD

Table 5 and Figure 16, which present the results of applying different percentages of NTD to gypseous soils under test, appear to have the impact of lowering the coefficient of permeability k values, especially at 0.3% and 0.5% of NTD for the three tested soils. For S1-tested soil, the reduction percentages of the k values were 79.02 and 80.0% when treated with 0.3 and 0.5% of NTD, respectively. While for S2 tested gypseous soil, the reduction percentages were 75.9 and 79.1%, and 66.04 and 73.6% for S3 tested gypseous soil when treated with 0.3 and 0.5% of NTD, respectively.

It is clear that adding 0.3% of NTD to the studied gypseous soils S1, S2, and S3 improves the coefficient of permeability k reasonably and cost-effectively compared with Al-Gharbawi [57], who obtained a 58.2% reduction in the coefficient of permeability k when 5% magnesium oxide was added to the tested sandy gypseous soil with a 47% gypsum content. Also, the obtained percentages of reduction, especially for S3 with high gypsum content in this study, were better and required less dosage than those obtained by Ali & Karkush [58], 41.8% when adding 2% of nanoclay to a soil with a gypsum content of 80%. Similarly, Emad & Salman [59] obtained a smaller reduction in the coefficient of permeability k 45% compared with the obtained result from the study when 3% of metakaolin was added to a gypseous soil with 54% gypsum content.

Table 5. Effect of NTD on the improvement ratios of k for the tested treated soils at the oedometer permeability leaching test

Soil type	Improvement ratio of the coefficient of permeability k			
	Untreated soil	0.1% of NTD	0.3% of NTD	0.5% of NTD
S1	-	53.7	79.02	80.0
S2	-	49.2	75.9	79.1
S3	-	45.3	66.04	73.6

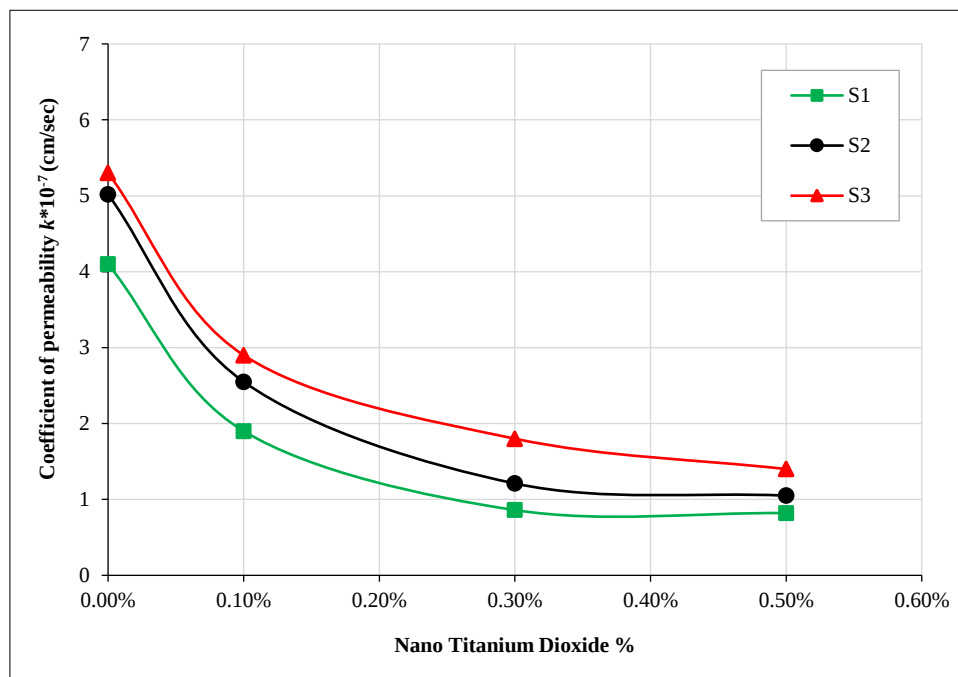


Figure 16. The coefficient of permeability versus nano titanium dioxide NTD

Figures 17 to 19 present the relationship between the leaching strain and the test time for the treated and untreated gypseous soil samples S1, S2, and S3 with varying NTD percentages, respectively. For the untreated gypseous tested soil samples, the leaching strain clearly increments with the test time, especially for the soils with the higher gypsum contents, S2 and S3. According to the strain-time behavior, the continuous dissolution of gypsum caused accordingly a continuous settlement.

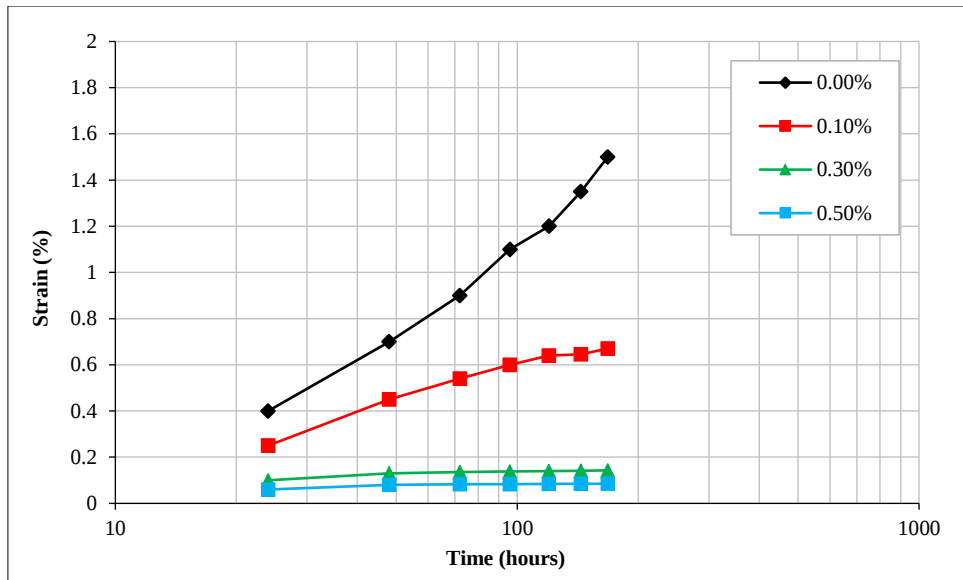


Figure 17. The leaching strain variation with time for S1 gypseous soil

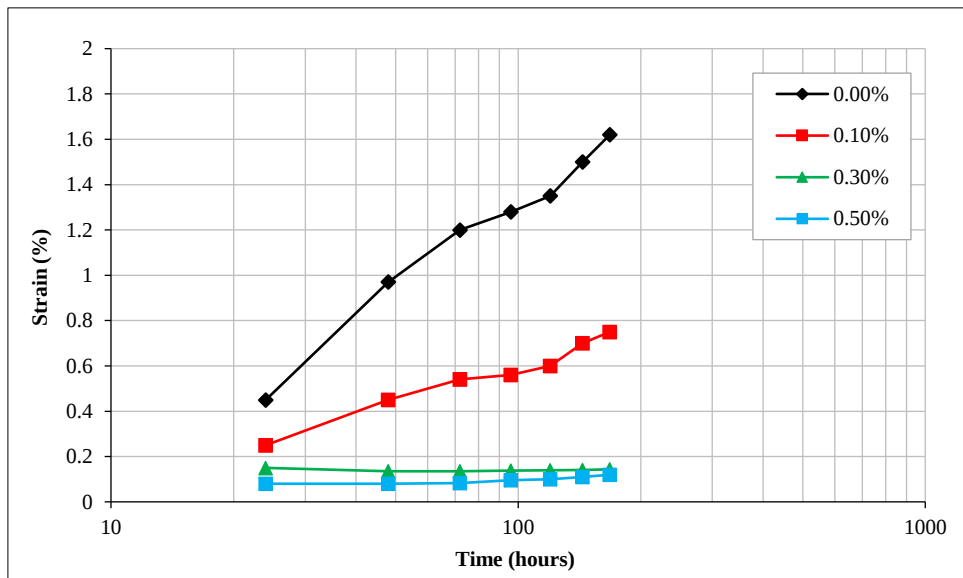


Figure 18. The leaching strain variation with time for S2 gypseous soil

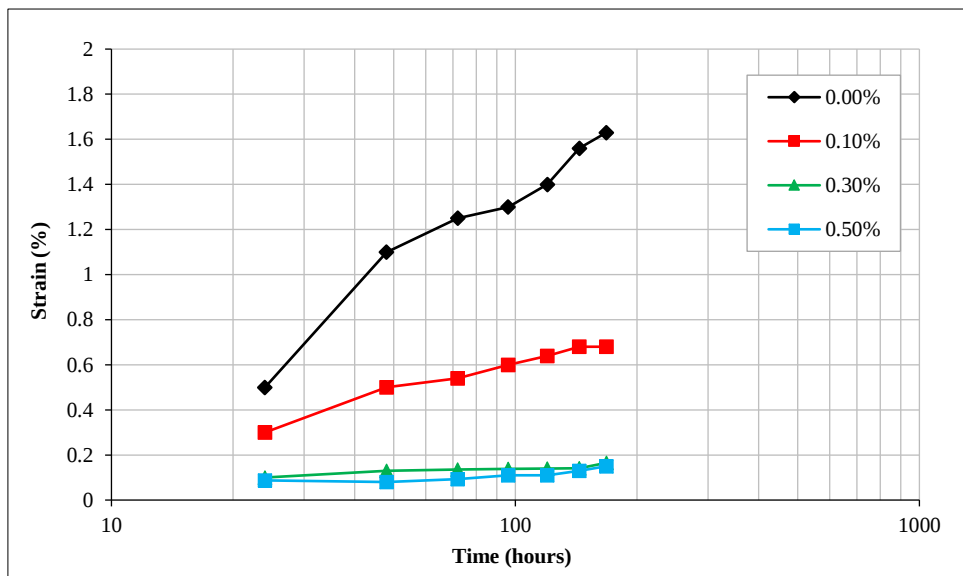


Figure 19. The leaching strain variation with time for S3 gypseous soil

The amount of leached salts, the kind of soil, the stress level, and the gypsum content all affect how much strain leaching occurs. The leaching of gypsum particles caused significant settlement [60]. The proportion of strain in the treated gypseous soil samples with NTD that were studied varies depending on the amount of leached gypsum and the percentage of NTD additive. However, this strain eventually ends after a certain amount of time. As the rate of the NTD increased, the leaching strain decreased and almost became constant, especially at the percentages 0.3 and 0.5% for the three tested gypseous soil samples S1, S2, and S3. It is demonstrated that applying 0.3% of NTD to the studied gypseous soils S1, S2, and S3 results in a fair and cost-effective improvement ratio for the leaching strain. For S1 tested soil, the percentage of reduction of the leaching strain was 90.5%, while for S2 and S3 tested soils, it was 91.2% and 89.9 %, respectively, when 0.3% of NTD was applied.

The difference in total dissolved salts TDS over time for the untreated and treated gypseous tested soil samples S1, S2, and S3 with 0.1, 0.3, and 0.5% of nano titanium dioxide NTD is displayed in Figures 20 to 22, respectively. Because of the continuous flow of water through the gypseous soils, which causes the soluble salts to dissolve and be removed, it is noted that for the untreated gypseous soils, the TDS in the leached water increased as the percentages of the gypsum content grew until the end of the test. This behavior matched the finding of Hassan Al-Riahi et al. [61], who concluded that the higher gypsum content in the soil led to an elevated TDS level. This phenomenon explains why the untreated gypseous soil samples show a considerable amount of leaching strain, which is larger than the initial settlement and has no definite endpoint upon the continuation of gypsum dissolution and leaching from the tested soil samples. The figures showed that the treated gypseous soils S1, S2, and S3 exhibited a decrease in the total dissolved TDS values as the percentages of the NTD increased. When 0.3% of NTD was added to the tested gypseous soil S1, the total dissolved salts TDS in the leached water reduced from 4.1 to 0.45 gm/l. while TDS for S2 and S3 reduced from 4.4 to 0.55 gm/l and 4.6 to 0.59 gm/l respectively. This tendency may be attributed to the effect of nano titanium dioxide, which coats the gypsum particles and isolates them partially or fully from the effect of flowing water.

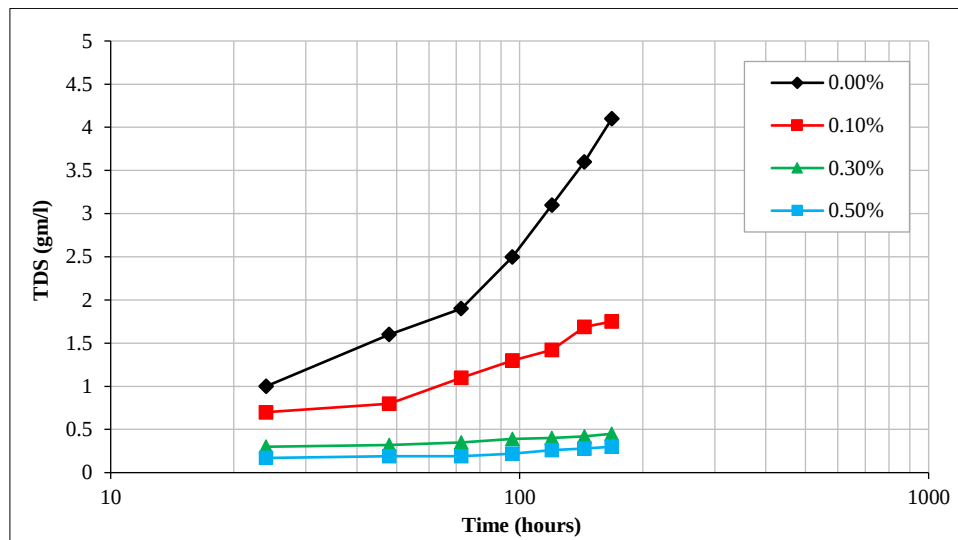


Figure 20. The total dissolved salts TDS versus the time of leaching for S1 gypseous soil

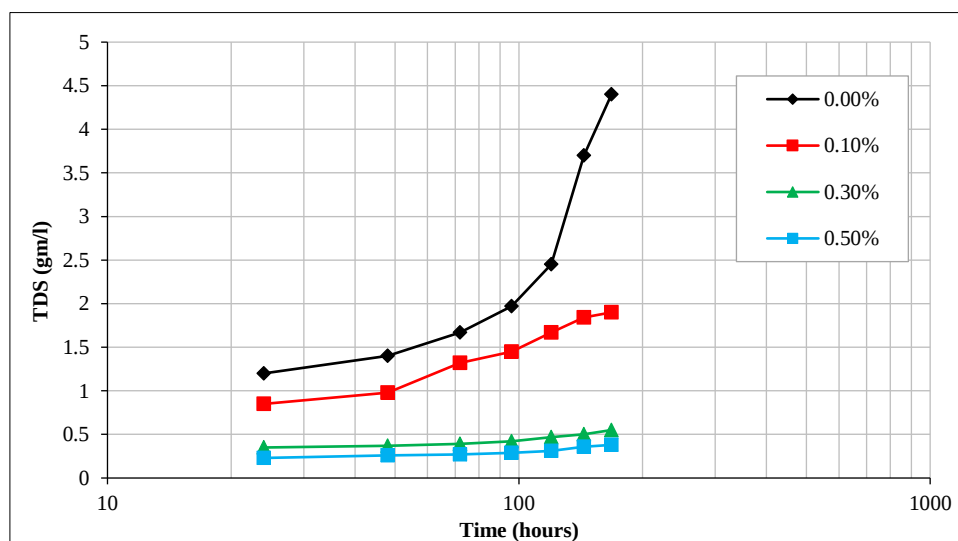


Figure 21. The total dissolved salts TDS versus the time of leaching for S2 gypseous soil

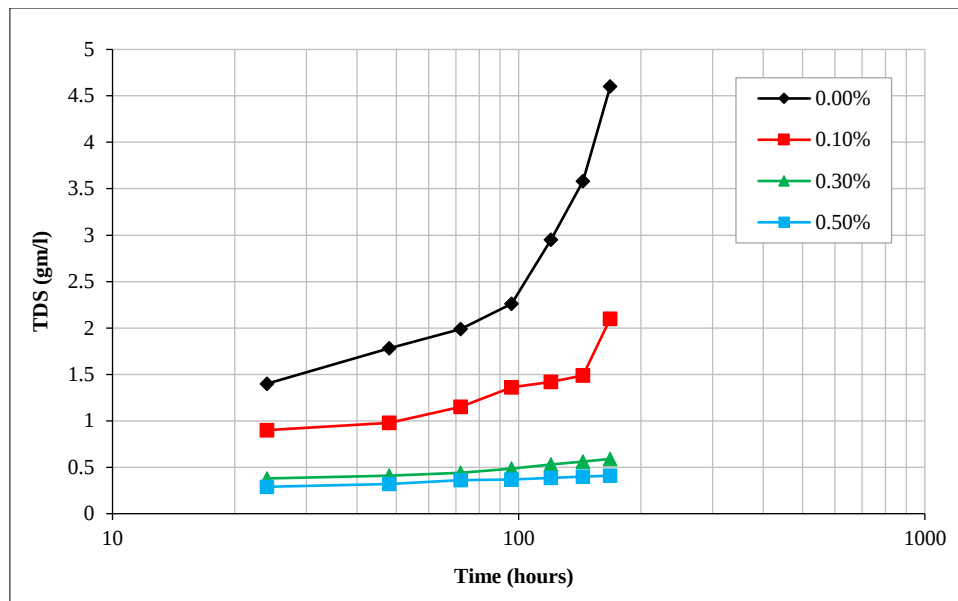


Figure 22. The total dissolved salts TDS versus the time of leaching for S3 gypseous soil

The reduction in the total dissolved salts TDS obtained in this work, especially after adding 0.3% of NTD, is much better than the reduction gained by Hussein et al. [60], where the total dissolved salts TDS dropped from 2.5 gm/l before treatment to 1.24 gm/l after adding 2% of pectin to the tested sandy gypseous soil with a 62% gypsum content.

It is revealed from Figures 23 to 25 that the pH values of the leached water for the three tested gypseous soils S1, S2, and S3 significantly increased with the continuous increase in the leaching time as a result of gypsum dissolving. This phenomenon can be attributed to the fact that water percolates through the soil during the leaching process, dissolving and carrying away soluble salts and other compounds. In the case that the leached water contains gypsum, the gypsum's dissolution may raise the concentration of calcium ions in the soil solution, neutralizing the acidic elements of the soil and raising the pH level.

A much more significant rise in the pH value was apparent for soil S3 than in the other soils. This is attributed to the presence of high gypsum content in site S3, which caused relatively high total dissolved salts TDS in the leached water from this gypseous soil. The pH values decreased as the percentages of the nano titanium dioxide NTD increased for S1, S2, and S3 due to the decreases in the TDS in the leached water, as shown in Table 6. It is clear that 0.3% of NTD gives a reliable pH value for the three tested soils.

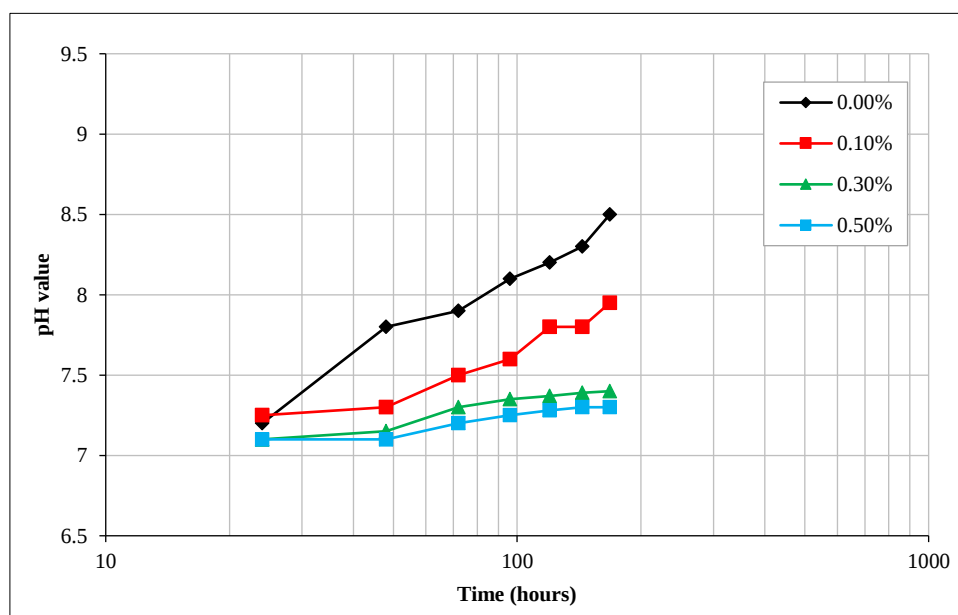


Figure 23. The pH value variation with time for S1 gypseous soil

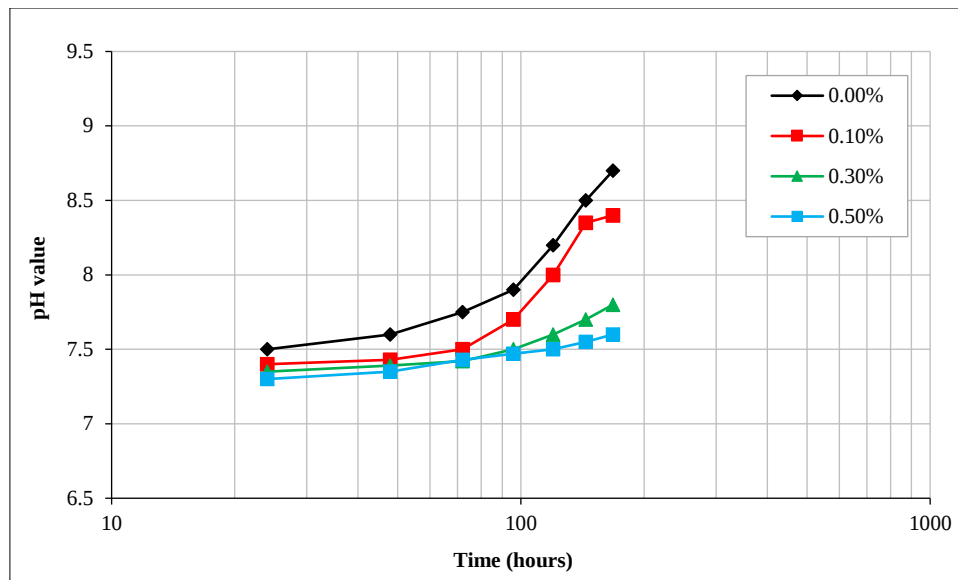


Figure 24. The pH value variation with time for S2 gypseous soil

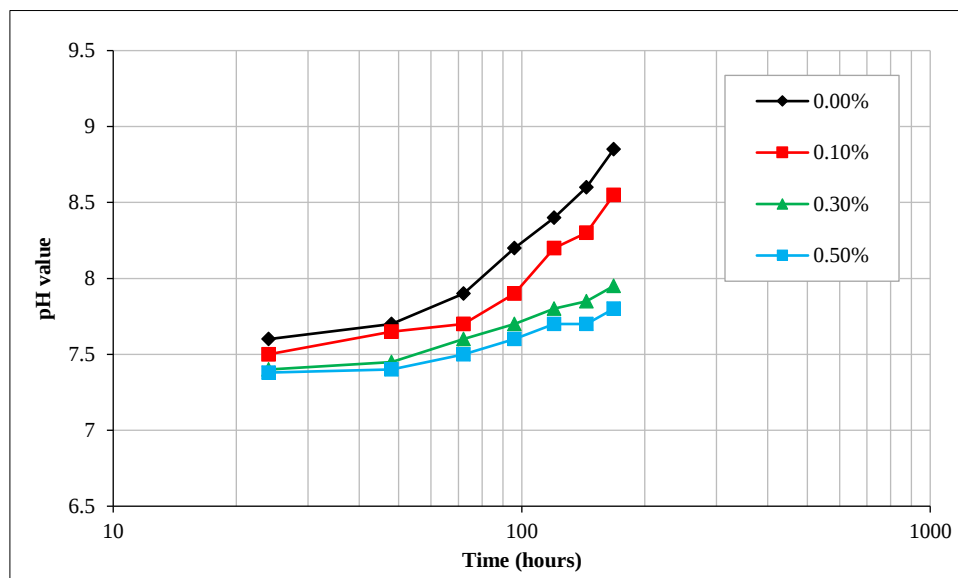


Figure 25. The pH value variation with time for S3 gypseous soil

Table 6. Effect of NTD on the pH values for the tested treated soils

Soil type	Improvement of the pH values			
	Untreated soil	0.1% of NTD	0.3% of NTD	0.5% of NTD
S1	8.5	8.0	7.4	7.3
S2	8.7	8.4	7.8	7.6
S3	8.9	8.6	8.0	7.8

4. Conclusions

The following conclusions could be obtained from experimental work:

- The oedometer permeability leaching test showed the significant effect of adding nano titanium dioxide (NTD) in different percentages on the coefficient of permeability (k), the leaching strain, the total dissolved salts (TDS), and the pH values of the three tested gypseous soils (S1, S2, and S3)
- The coefficient of permeability k increased sharply with time for the untreated tested gypseous soils S1, S2, and S3, especially at the beginning of the test, and then continuously decreased in small amounts until the end of the test. This behavior may be attributed to the collapse of soil particle structure, conjugate with the continuous removal of cementing material (gypsum) due to the leaching process. The results of the tested gypseous soils

treated with different percentages of NTD showed an apparent effect on reducing the values of the coefficient of permeability k , especially at 0.3 and 0.5% of NTD for the three tested soils. Adding 0.3% of NTD to the studied gypseous soils S1, S2, and S3 improves the coefficient of permeability k reasonably and cost-effectively at about 79.02, 75.9, and 66.04, respectively.

- For the untreated gypseous soil during the leaching process, the leaching strain increased with time as the leaching process continued. The strain rate for the improved soils was slightly increased with the time of the test, especially at the 0.3 and 0.5% percentages compared with the 0.1% of the additive for the three gypseous tested soil samples S1, S2, and S3. The leaching strain in gypseous soil S1 was less than the strain in gypseous soils S2 and S3. The strain increased as the total dissolved salt increased. The treated gypseous soils are exposed to less gypsum dissolution, as the nano titanium dioxide (NTD) material forms an impermeable layer to prevent direct contact between water and gypsum. This reduces gypsum dissolution and, thus, reduces leaching strain. For S1 tested soil, the percentage of reduction of the leaching strain was 90.5%, while for S2 and S3 tested soils, it was 91.2% and 89.9%, respectively, when 0.3% of NTD was applied.
- The amount of total dissolved salts (TDS) increased significantly in the leached water during the oedometer permeability leaching test for the gypseous tested soil samples S1, S2, and S3. However, TDS decreased as the percentage of the NTD increased for the three gypseous tested soils; especially at 0.3 and 0.5%, the values of TDS became almost constant. When 0.3% of NTD was added to the tested gypseous soil S1, the total dissolved salts (TDS) in the leached water reduced from 4.1 to 0.45 gm/l, while TDS for S2 and S3 reduced from 4.4 to 0.55 gm/l and 4.6 to 0.59 gm/l, respectively. This trend may be attributed to the effect of nano titanium dioxide, which coats the gypsum particles and isolates them partially or fully from the effect of flowing water.
- With the continuous increase in the leaching time, the pH values of the leached water for the three tested gypseous soils significantly increased due to gypsum dissolving. A much more significant rise in pH value was apparent for soil S3 than in the other soils. This is attributed to the presence of high gypsum content in site S3, which caused relatively high total dissolved salts (TDS) in the leached water from this gypseous soil. As the percentage of the nano titanium dioxide (NTD) increased for S1, S2, and S3, the pH values decreased due to decreased TDS in the leached water. It is clear that 0.3% of NTD gives a reliable pH value for the three tested soils.

5. Declarations

5.1. Author Contributions

Conceptualization, N.W.J. and M.A.; methodology, N.W.J. and M.Y.F.; investigation, N.W.J. and M.A.; data curation, N.W.J.; writing—original draft preparation, N.W.J.; writing—review and editing, M.A. and M.Y.F.; visualization, N.W.J.; supervision, M.A. and M.Y.F.; funding acquisition, N.W.J. All authors have read and agreed to the published version of the manuscript.

5.2. Data Availability Statement

The data presented in this study are available in the article.

5.3. Funding

The authors received no financial support for the research, authorship, and/or publication of this article.

5.4. Conflicts of Interest

The authors declare no conflict of interest.

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