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Effect of nano-stabilizer on geotechnical properties of leached gypsiferous soil

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Abstract. Gypsiferous soils classified as problematic soils due to the dissolution of gypsum. Presence of gypsum in the soils texture subjected to steady flow can cause serious damages for the buildings, roads and water transmission canals. Therefore, researchers have conducted a series of physical, mechanical and microstructural laboratory tests to study the effect of gypsum leaching on the geotechnical properties of a lean clay containing 0%, 3%, 6%, 9%, 12%, and 15% raw gypsum. In addition, a combination of two nano-chemical stabilizers named Terrasil and Zycobond was used in equal proportions to stabilize the gypsiferous clayey samples. The results indicated that gypsum leaching considerably changed the physical and mechanical properties of gypsiferous soils. Further, adding the combination of Terrasil and Zycobond nano-polymeric stabilizers to the gypsiferous soil led to a remarkable reduction in the settlement drop, compressibility, and electrical conductivity (EC) of the water passing through the specimens, resulting in improving the engineering properties of the soil samples. The X-ray diffraction patterns indicate that stabilization by terrasil and zycobond causes formation of new peaks such as CSH and alteration of pure soil structure by adding raw gypsum. Scanning electron microscope (SEM) images show the denser texture of the soil samples due to chemical stabilization and decrease of Si/Al ratio which indicates by Energy dispersive X-ray (EDS) interpretation, proved the enhance of shear strength in stabilized samples.

Keywords: gypsiferous soil; nano-stabilization; terrasil; zycobond; dissolution; microstructure

1. Introduction

Gypsiferous soils, which are spread in arid and semi-arid areas in the world, such as Iran, Iraq, Arabian Peninsula, Armenia, United States, Spain and Russia (Boyadgiev and Verheye 1996, Casby-Horton *et al.* 2015, Schanz and Karim 2018), may have relatively acceptable engineering properties in dry mode. However, the penetration of stable leakage flow to the texture of gypsiferous soils results in the gradual dissolution of the gypsum, and consequently, the loss of inter-particle cementation, which leads to the significant weakening of geotechnical parameters including porosity, compressibility, hydraulic conductivity, and the collapse of the soil structure (Ahmad *et al.* 2012, Fattah *et al.* 2012, Kuttah and Sato 2015, Karim *el al.* 2017). Therefore, the gypsum dissolution generally increases the collapsibility of the soil, which, in turn, leads to unpredictable settlements. The settlement of the gypsiferous soils due to the gypsum dissolution can result in failure of various structures constructed on the gypsiferous soil deposits such as buildings, dams, roadbeds and water transmission canals (Namiq and Nashat 2011). Thus, the gypsiferous soils are classified as problematic soils and it is essential to study about more effective gypsiferous soil improvement methods.

Soil stabilization defined as the procedure of enhancing shear strength parameters and decreasing the permeability and compressibility of the soil (Shooshpasha and Alijani Shirvani 2015). Generally, soil stabilization approaches can be classified into mechanical and chemical methods. Most mechanical methods improve the soil characteristics by compaction, while chemical methods enhance inter-particle bonds through cementation by adding chemical additives to the soil (Qureshi *et al.* 2017). Chemical improvement has been used by many researchers for improving the performance of the soils (Kalantari *et al.* 2010, Chang *et al.* 2015, Canakci *et al.* 2015, Mirzababaei *et al.* 2018, Taha *et al.* 2018, Kwon *et al.* 2019). The stabilization of the gypsiferous soils can be a suitable option to prevent from the damages caused by the gypsum dissolution to the hydraulic structures such as water transmission canals. Researchers have conducted numerous studies over recent years to improve the engineering properties of fine- and coarse-grained gypsiferous soils, using various chemical and mechanical methods. The use of chemical additives such as lime (Al-Zubaydi 2011, Jha and Sivapullaiah 2016b), cement (Awn *et al.* 2012), asphalt (Taha *et al.* 2008, Kadhim 2014), ceramic (Al-Numani 2010), crude oil (Aziz and Ma 2011), rice husk ash (Alateya 2013), calcium chloride (Ibrahim *et al.* 2016), fly ash (Alsafi *et al.* 2017, Jha and Sivapullaiah 2018), silicone oil (Ibrahim and Schanz 2017), geosynthetics (Karim 2017) and silica fume (Moayyeri *et al.* 2019), and the application of mechanical methods such as static compaction (Razouki *et al.* 2012, Razouki and Ibrahim 2017), stone column (Al-Obaidy *et al.* 2016, Karim *et al.* 2016), grouting (Fattah *et al.* 2014) and dynamic compaction (Fattah *et al.* 2012, Al-Layla and

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Alsaffar 2014) are among the common measures taken in the past years, in order to stabilize the gypsiferous soils. However, a significant portion of recent researches are unable to conduct an operational and comprehensive overview on the stabilization of gypsiferous soils methods to practically address their efficiency, due to the technical limitations, environmental damages, and considerable costs applied to the project during the construction periods. For example, most research has mainly focused on the use of traditional materials such as lime, cement and petroleum products in the stabilization of gypsiferous soil, which has environmental contaminations and high transmission costs, causing serious challenges in the justification of the projects.

Terrasil is a water-soluble, non-leachable, cost effective and environmental friendly compound (Aderinola and Nnochiri 2017, Singh 2019, Meeravali *et al.* 2020). As an organosilane compound which is resistant to heat and UV radiation, Terrasil reacts with the soil particles and forms permanent and stable nano-siliconized hydrophobic surfaces by converting the hydrophilic silanol groups to the siloxane hydrophobic bonds (Aderinola and Nnochiri 2017, Selvaraj *et al.* 2018). This phenomenon makes the soil particles more resistant to water and results in appropriate conditions for the soil compaction. Terrasil plays an effective role in improving different types of fine grained and coarse grained soil (Patel *et al.* 2015b, Thomas *et al.* 2016, Aderinola and Nnochiri 2017, Ravi Shankar and Panditharadhya 2017, Rathod 2017, Kalyani *et al.* 2018). The resistance to heat and UV radiation is regarded as one of the most important characteristics of the Terrasil, which justifies the use of this polymeric nano-stabilizer in scorching areas like Khuzestan province (case study). In addition, the waterproofing characteristic of the Terrasil is another important factor that persuades the researchers to use it for improving gypsiferous soils existing in the embankment of water transmission canals, which always subject to the stable leakage flow that accelerates the rate of gypsum dissolution. Zycobond is an acrylic nano-polymer with the particles smaller than 90 nm, which increases the soil strength against erosion and failure by being present in the soil mass and forming strong bonds between the soil particles (Patel *et al.* 2015b, Padmavathi *et al.* 2018). Terrasil as a waterproof material and Zycobond as a bonder one, is well known in chemical industries and the soil stabilization resulted from the combination of these two nano-materials prevents from both the leaching and the breakage of bonds between the soil particles (Mulla and Guptha 2019). The addition of these two nanomaterials results in considerable changes at the atomic and molecular levels of the soil particles, resulting in improving the geotechnical parameters of the soil, such as strength and permeability (Raghavendra *et al.* 2018).

By adding 0.041% Terrasil to a CL soil collected from an area in India, Patel *et al.* (2015a) concluded that the presence of Terrasil in the soil could reduce the plasticity index and permeability. He also found that the values of CBR for the samples stabilized with Terrasil are significantly higher than those for the unstabilized ones. Mrudul *et al.* (2016) conducted gradation, standard proctor compaction, Atterberg limits and CBR tests to study the effect of adding Terrasil and cement to a blackish silty clay with medium plasticity (MI) on the engineering properties of the soil. The results revealed that the addition of Terrasil and cement to the soil in weight percent of 1 and 0.04% would give the highest CBR, respectively. They also concluded that using cement and Terrasil for stabilizing road subgrades would produce acceptable engineering results and lead to significant savings on the project costs. By studying the effect of stabilizing a CH soil with lime (2 wt.%) and Terrasil (0.05, 0.07 and 0.09wt.%), Pandagre and Jain (2017) observed that adding 0.07% Terrasil to the tested soil would give the highest values of CBR and unconfined compressive strength after three weeks of curing. Olaniyan and Ajileye (2018) investigated the geotechnical properties of two fine-grained soil samples collected from an area in Nigeria and stabilized with nano-chemicals (Terrasil and Zycobond) by weight percent of 5, 10, 15 and 20%. He found that the addition of this nano-chemical stabilizer to the soil increases the plastic and liquid limits and the optimum moisture content while reducing the maximum dry density. Moreover, the values of CBR for the stabilized samples showed remarkable improvement compared to those for unstabilized ones. The highest values of strength parameters were obtained by adding 15% nano stabilizer to the soil.

2. Materials and methods

The pure clay and the clay containing 15% raw gypsum used in this study were collected from Arayez plain in Khuzestan province located in the southwest of Iran. Table 1 presents the physical characteristics of the pure clay. Figs. 1, 2 and 3 illustrate the gradation curve, the SEM (Scanning Electron Microscope) image and the XRD (X-ray Diffraction) graph of the pure clay used in the research, respectively.

Terrasil used in this study was prepared from Zydex Company in Gujarat, India. It is a pale yellow liquid which forms a clear solution by mixing with water and its density is 1.01 gr/cm^3 (Johnson and Rangaswamy 2015, Pandagre and Jian 2017). Among the ingredients of this nano-chemical stabilizer, benzyl alcohol (25%-27%) acts as a solvent and causes the adherence of the stabilizer solution to the surfaces of the particles, ethylene glycol (3%-5%) keeps the ambient temperature constant during the reaction and Hydroxyalkyl-alkoxy-alkysiyl compound (65%-70%) acts as a binder, emulsion stabilizer, and film former (Ewa *et al.* 2016, Singh *et al.* 2017b). Terrasil is available in the form of a concentrated liquid, which is mixed with water before mixing with the soil. Zycobond, which is a nano-polymer and can bond the soil particles to each other, was bought from Zydex Company in Gujarat, India. This nano-stabilizer is a milky white sticky liquid that soluble in water (Rohith *et al.* 2018).

To prepare the unstabilized laboratory samples, the pure soil (containing 0% gypsum) and base gypsiferous soil (containing 15% raw gypsum) were passed through sieve No. 40 (0.425 mm) and stored at 60°C for 48 h to evaporate the excess moisture content of the materials (Aldaood *et al.* 2014). The above temperature setting used at the end of

Table 1 Physical characteristics of the pure soil

G_s	LL (%)	PL (%)	PI (%)	OMC (%)	MDD (g/cm^3)	USCS
2.70	34	18.5	15.5	11.3	1.70	CL

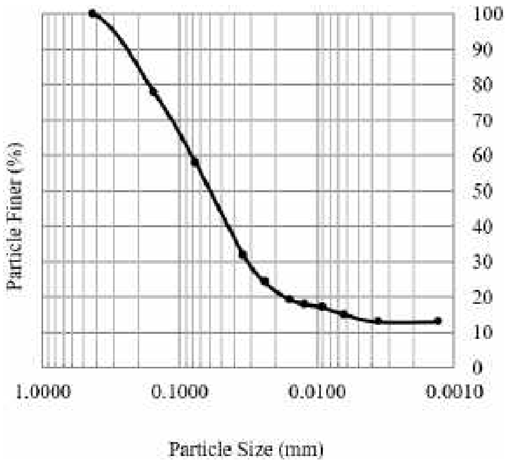

Fig. 1 Grading curve of the pure soil

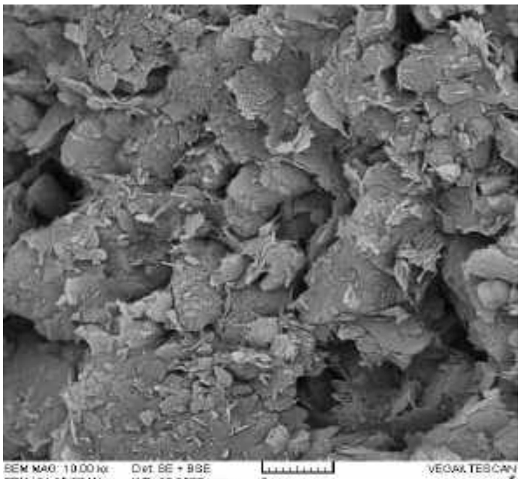

Fig. 2 SEM image of the pure soil

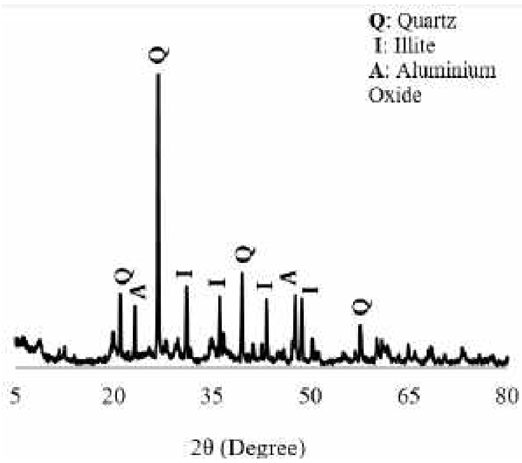

Fig. 3 XRD graph of the pure soil

different experiments to dry the specimens was chosen to prevent the evaporation of the gypsum crystallisation water

Table 2 Laboratory tests and microstructural analysis

Laboratory Test	**Standard / Specification**
Sieve Analysis	ASTM D6913
Hydrometer Analysis	ASTM D521 – 58
Unified Soil Classification System	ASTM D2487 – 17
Specific Gravity (G_s)	ASTM D854-14
Standard Proctor Compaction	ASTM D698-12
Atterberg Limits	ASTM D4318-87
Modified consolidation	----------
Direct Shear	ASTM D3080
EC (electrical conductivity)	----------
X-ray Diffraction (XRD) Analysis	XRD analysis with Asenware AW-DX300 Apparatus, Cu/Kα radiation, 2°/ min scan speed
SEM analysis and EDS interpretation	SEM analysis and EDS with Tescan Vega-II XMU apparatus on micro level structure of soil specimens

that have notable effect on physical, mechanical and mineralogical properties of raw gypsum (Arakelyan 1986). In the next step, specimens containing 3%, 6%, 9% and 12% raw gypsum were prepared by blending pure soil and gypsiferous soil containing 15% gypsum in different ratios. Then, the mixtures were blended with a planetary ball mill at 350 rpm for 15 min to prepare uniform specimens. After that, by using standard proctor compaction test, the maximum dry density (MDD) and optimum moisture content (OMC) values determined for specimens containing different raw gypsum content. At the last preparation stage, water was added to the various mixtures of soil and raw gypsum to achieve optimum moisture content. To uniformly distribute the moisture, the soil specimens were placed within plastic bags for 24 hours (Kargar *et al.* 2014). All of the steps implemented to make the stabilized samples were similar to the method used for making unstabilized ones, except that the addition of stabilizer to the clay soil samples contained various percentages of raw gypsum. Terrasil and Zycobond, as the two nano-chemical stabilizers were solved at the same proportion in one liter per ton weight of the soil and after dissolving in water (at OMC level), sprayed to the soil samples (Singh 2017a). In order to reach the maximum efficiency, the tests were carried out 72 hours after the addition of the solution containing nano-chemical stabilizers to the soil samples. Laboratory tests and microstructural analysis which used in this study, listed in Table 2. In the modified consolidation test, soil compacted in the standard proctor compaction apparatus at OMC (optimum moisture content) level and sampling was done from the middle part of the compacted soil by use of consolidation ring. Then, the loads of 25, 50, 100, 200, 400, and 800 kPa applied to the specimens, independently. The water remain in the consolidation apparatus cup was substituted by the fresh water (EC=0) every day for 15 days and the electrical conductivity (EC) of water passing through the specimens and the settlement of the specimens (S) were measured at time intervals of 24 hours for 15 days for unstabilized and stabilized samples. The difference between the modified consolidation test used in this study and the standard consolidation test is that in the modified method, each sample is exposed to a single stress level for 15 days to evaluate the effect of stress on the gypsum dissolution, independently which is required to model the field conditions. In the standard consolidation test, due to continuous and connected loading, the impact of applied stress on the gypsum dissolution is not measurable. The strength parameters of stabilized and unstabilized samples which wetted to the optimum moisture content (OMC) level were calculated from the direct shear test and Mohr-Coulomb failure envelope. In the direct shear test procedure, the normal stresses of 25, 50, 100, 200, 400, and 800 kPa applied to the soil samples and the shearing rate set on 1 mm/min. Moreover, the microstructural examinations including XRD (x-ray diffraction) graphs, SEM (scanning electron microscope) images and energy-dispersive x-ray spectroscopy (EDS) interpretations were conducted on the unstabilized and stabilized samples with Terrasil and Zycobond solutions.

At a particular percentage of gypsum and by applying a specified stress, the settlement drop (ΔS) of the stabilized and unstabilized samples due to gypsum presence and gypsum dissolution can be obtained by the following equation:

$$\Delta S = S_{tgs} - S_{tps} \tag{1}$$

where, S_{tgs} represents the total settlement of the sample and S_{tps} illustrates the total settlement of pure clay (soil containing 0% gypsum). Also, by considering the dissolution electrical conductivity of pure clay samples equal to zero, the parameter EC_{dgs} for a constant applied stress level and a particular percentage of gypsum is obtained from the Eq. (2):

$$EC_{dgs} = EC_{tgs} - EC_{tps} \tag{2}$$

where, EC_{dgs} demonstrates the dissolution electrical conductivity of the water passing through the gypsiferous sample, EC_{tgs} indicates the total electrical conductivity of water passing through the gypsiferous sample and EC_{tps} represents the total electrical conductivity of water passing the pure clay. At specified gypsum contents, the values of the cohesion loss (Δc) due to gypsum presence and gypsum dissolution for stabilized and unstabilized gypsiferous soil samples are obtained by the following equation:

$$\Delta c = c_{tps} - c_{tgs} \tag{3}$$

where, c_{tps} indicates the cohesion of pure clay (soil containing 0% raw gypsum), c_{tgs} is the cohesion of gypsiferous soil sample.

3. Results

Table 3 provides changes in some physical properties of the gypsiferous clay samples for different percentages of raw gypsum. As observed, increase in the amount of raw gypsum in the soil mass, decreases the samples' consistency limits such as plastic limit (PL), liquid limit (LL), and plasticity index (PI), significantly. This phenomenon can be due to the replacement of soil monovalent ions with bivalent calcium ions existing in the gypsum on the one hand, and the much lower density of gypsum compared to the soil on the other hand (Jha and Sivapullaiah 2016a). The second reason, which is the considerable difference between the G_s of soil and gypsum, causes a reduction in the densities of the sample by increasing the percentage of raw gypsum in the soil.

Table 3 also indicates the values of two major compaction characteristics of the soil samples containing different percentages of raw gypsum, i.e., the optimum moisture content (OMC) and the maximum dry density

Table 3 Index Properties of soil-gypsum mixtures

Gypsum Content (%)	Compaction Characteristics		Atterberg Limits		
	MDD (gr/cm^3)	OMC (%)	LL (%)	PL (%)	PI (%)
0	1.70	11.3	34	18.5	15.5
3	1.69	11.8	33.1	17.9	15.2
6	1.68	12.5	30	16.1	13.9
9	1.66	13.1	28.9	15.8	13.1
12	1.63	14.4	27.3	14.3	13
15	1.59	15.7	24.9	13.7	11.2

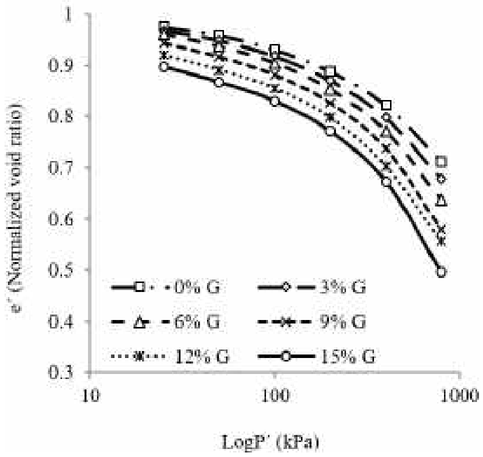

Fig. 4 Normalized void ratio (e′) versus logarithmic effective stress of soil-gypsum mixtures with different gypsum contents (unstabilized samples)

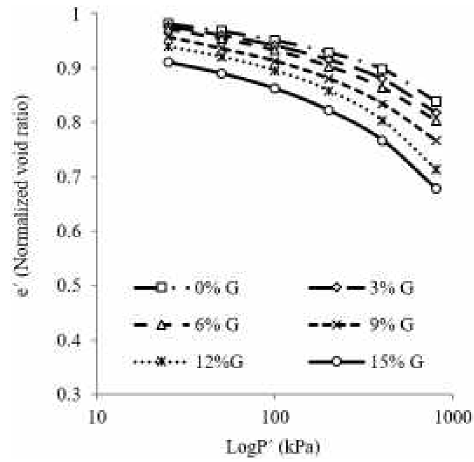

Fig. 5 Normalized void ratio (e′) versus logarithmic effective stress of soil-gypsum mixtures with different gypsum contents (stabilized samples)

(MDD). Accordingly, increasing gypsum content enhances the optimum moisture content and decreases the maximum dry density. The reason for the increase in the optimum moisture content is the larger specific surface area (SSA) of gypsum particles compared to the soil (SSA_{Soil}= 80.5 m^2/g, SSA_{Gypsum}= 138 m^2/g), leading to the higher tendency of gypsum to absorb the moisture. In addition, the reduction in the maximum dry density is attributed to the significant difference between the specific gravity (G_s) of raw gypsum and the soil ($G_{s(soil)}$= 2.70 and $G_{s(gypsum)}$ =2.33), resulting in reducing the density of the sample by increasing the amount of gypsum in a constant volume of the soil.

Figs. 4 and 5 depict e′-Log P′ graphs for the lean clay soil containing 0%, 3%, 6%, 9%, 12% and 15% of raw gypsum for unstabilized and stabilized samples, respectively. The normalized void ratio (e′) calculated by dividing final void ratio (e_f: void ratio at the end of the modified consolidation test) by initial void ratio (e_0: void ratio before modified consolidation test). At a constant applied stress level, increasing the raw gypsum content reduces the normalized void ratio ($e' = e_f/e_0$) in both unstabilized and stabilized specimens while increasing the stress applied to the samples at a constant percentage of gypsum reduces the e′.

These observations can be attributed to the finer size of gypsum particles compared to that of clay aggregates which leads to the formation of more pores with larger volumes, resulting in increasing the porosity and settlement in a constant volume of the soil. On the other hand, the dissolution removes a considerable amount of gypsum from the soil texture, which, in turn, increases the normalized void ratio. Therefore, it can be concluded that increase in the gypsum content and the applied stress causes the dissolution of more gypsum, reduces the values of e′ and consequently, e′-LogP′ curves become diverged from each other. Furthermore, it is observed that the normalized void ratio (e′) is much lower for the stabilized samples compared to the unstabilized ones and this difference is increased more by raising the gypsum content and the applied stress. In addition, increasing the gypsum percentage and the applied stress leads to fewer changes in the values of normalized void ratio for stabilized samples compared to unstabilized ones. As illustrated, the graphs of Fig. 5 are much closer together than that of the graphs shown in Fig. 4, meaning that the Terrasil and Zycobond reduce the gypsum dissolution in the 15-days period, significantly.

Fig. 6 shows the values of characteristics related to the compressibility behavior of the clay samples with low plasticity, containing 0%, 3%, 6%, 9%, 12% and 15% of raw gypsum for the unstabilized samples and samples stabilized with nano-chemical materials. As demonstrated, in all of the samples, including the stabilized and unstabilized ones, increasing the gypsum content of the soil samples and gypsum dissolution phenomenon increase the values of C_c (compression index) and k (permeability coefficient), considerably. For example, by increasing gypsum content up to 12% in unstabilized samples, the compression index (C_c) increases from 0.103 to 0.151. In other words, the compression index of the specimen increases by approximately 47% in this case. Regarding the

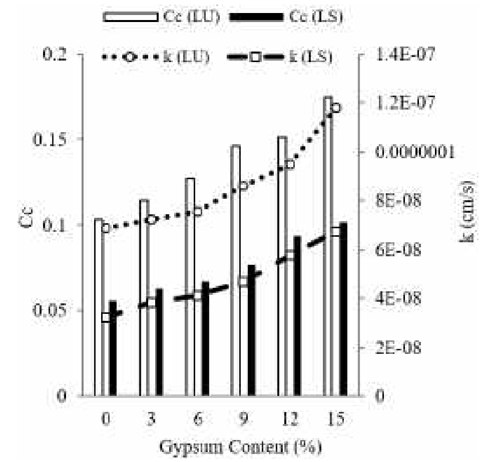

Fig. 6 Compression index (C_c) and permeability coefficient (k) of Soil blended with 3% and 15% raw gypsum for leached-unstabilized (LU) and leached-stabilized (LS) specimens

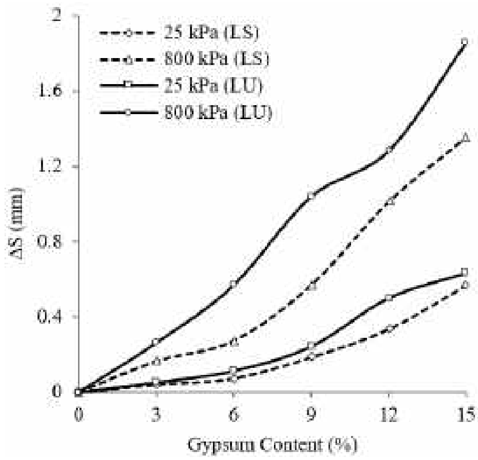

Fig. 7 Settlement drop curves for leached-unstabilized (LU) and leached-stabilized (LS) soil samples blended with 3% and 15% gypsum under 25 and 800 kPa applied stresses

stabilized specimens, adding 12% raw gypsum to the soil increases the coefficient of permeability from $3.24*10^{-8}$ to $5.77*10^{-8}$ cm/s. Thus, the soil permeability is increased by approximately 78%.

On the other hand, it is observed that adding nano-materials to the clay soil containing different percentages of gypsum decreases the compressibility parameters (C_c and k) greatly and therefore, improves the engineering properties of lean clay soil. For instance, stabilization of soil specimen containing 15% raw gypsum by nano-materials reduces the compression index (C_c) from 0.175 to 0.101and decreases the coefficient of permeability (k) from $1.18*10^{-7}$ to $0.673*10^{-7}$ cm/s, respectively. In the other words, by stabilization, the values of compression index and permeability coefficient reduced 73 and 75 percent, respectively and thus, adding Terrasil and Zycobond to gypsiferous clayey soil samples causes effective changes in compressibility parameters.

Fig. 7 demonstrates the graphs of settlement drop (ΔS) of the unstabilized and stabilized samples containing 0%,

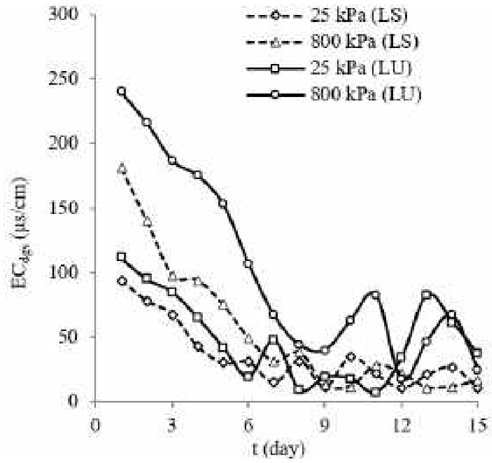

Fig. 8 Dissolution electrical conductivity (EC_{dgs}) curves for leached-unstabilized (LU) and leached-stabilized (LS) samples containing 3% gypsum under 25 and 800 kPa applied stresses

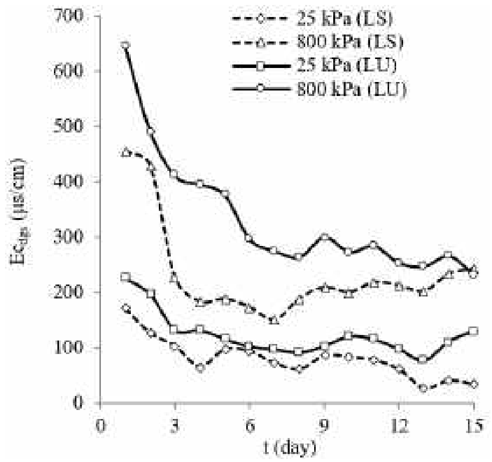

Fig. 9 Dissolution electrical conductivity (EC_{dgs}) curves for leached-unstabilized (LU) and leached-stabilized (LS) samples containing 15% gypsum under 25 and 800 kPa applied stresses

3%, 6%, 9%, 12% and 15% raw gypsum. It can be seen that by increasing the amount of raw gypsum in the soil samples and also, by increasing the applied stress at constant gypsum contents, the settlement drop of the specimens increased, remarkably. In other words, an increase in the amount of gypsum and the applied stress in the samples with the same gypsum percentage leads to an increase in the gypsum in the settlement of the soil samples. Generally, increasing the amount of gypsum content raises the slope of the settlement drop of the gypsiferous clay samples curve. Based on the results obtained from the modified consolidation tests, it was also concluded that the samples stabilized with nano-materials experienced lower settlements compared to unstabilized ones. For example, for the clay sample containing 9% gypsum, which was subjected to a stress of 25 kPa, the stabilization of gypsiferous soil with Terrasil and Zycobond reduces the settlement drop of the sample from 0.243 to 0.186 mm, indicating a reduction of almost 31%. The changes in the settlement drop of the stabilized samples are higher for the

Table 4 Cohesion loss (Δc) of the leached- stabilized (LS) and leached - unstablized (LU) soil samples containing 0%, 3%, 6%, 9%, 12%, and 15% gypsum

Gypsum Content (%)	Δc (kg/cm^2)	
	LS Samples	LU Samples
0	0.00	0.00
3	0.03	0.12
6	0.08	0.19
9	0.24	0.26
12	0.38	0.53
15	0.46	0.69

samples under higher applied stresses compared to unstabilized ones. Stabilization of gypsiferous soil with nano-materials has changed the settlement drop from 1.040 to 0.569 mm in the sample containing 9% gypsum under a surcharge of 800 kPa, indicating a reduction of almost 82% in the settlement drop. This trend implies that the combination of Terrasil and Zycobond decreases the gypsum dissolution, significantly.

Figs. 8 and 9 provide the dissolution electrical conductivity of water passing through the gypsiferous sample (EC_{dgs}) graphs in a 15-days period for the stabilized and unstabilized specimens under the stresses of 25 and 800 kPa, containing 3% and 15% gypsum. As shown in the figures, owing to the considerable amounts of soluble salts within the soil texture, the values of the EC_{dgs} are high during the early stage of the leaching process for both stabilized and unstabilized gypsiferous soil samples. Over time, given the gradual dissolution of gypsum, the EC_{dgs} values decrease remarkably and eventually, they tend a constant value. On the other hand, at constant gypsum content and under a constant applied stress, the samples stabilized with nano-materials consisting of a mixture of Terrasil and Zycobond show lower dissolution electrical conductivities compared to unstabilized ones. Thus, the nano-chemical stabilizers used in the present study play an important role in improving the engineering properties of the lean clay containing different percentages of gypsum, by reducing the gypsum leaching and consequently preventing the soil structure from being more porous and weaker.

By increasing gypsum content of the unstabilized and stabilized samples, the EC_{dgs} increased, indicating that increasing the amount of gypsum in the soil increases the gypsum dissolution. It is also observed that by increasing the applied stress level, the dissolution electrical conductivity of the samples increased, explaining that increasing the stress leads to the dissolution of more amount of gypsum. For both stabilized and unstabilized samples, the gradient of the changes in the dissolution electrical conductivity graphs of the samples containing 15% gypsum is more than that of the similar samples containing 3% gypsum, showing an increase in the dissolution rate through increasing the amount of gypsum. Furthermore, the trend of the changes in the dissolution electrical conductivity of the stabilized samples is generally more regular compared to

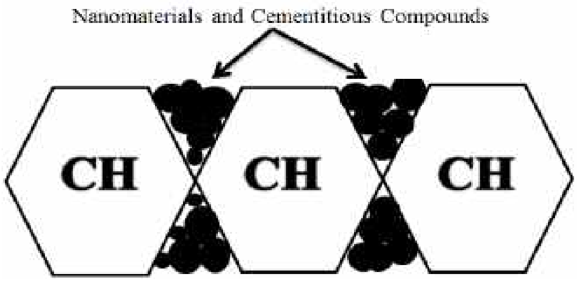

Fig. 10 Effect of nano-materials and cementitious compounds on filling internal pores formed by CH

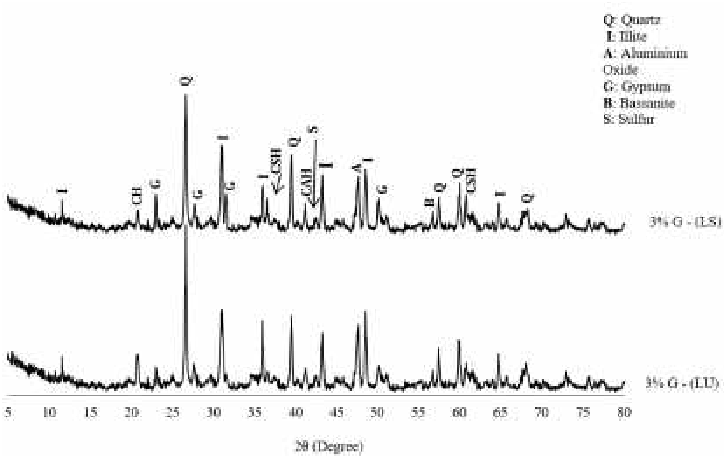

Fig. 11 XRD analysis curves for leached-unstabilized (LU) and leached-stabilized (LS) soil samples blended with 3% gypsum

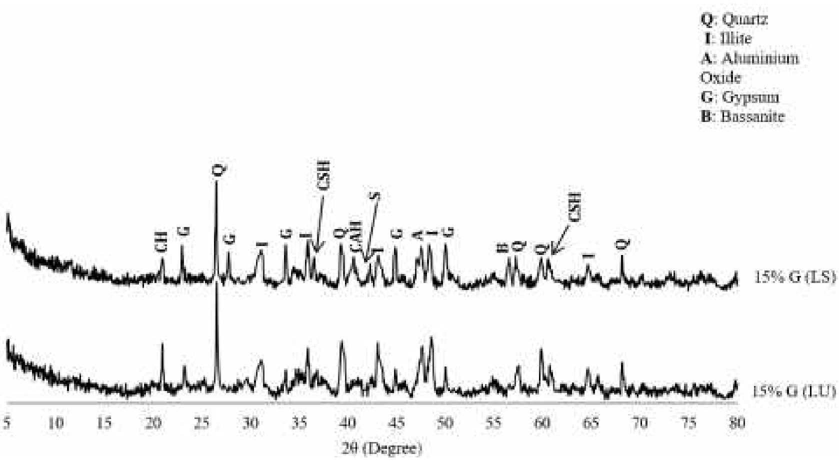

Fig. 12 XRD analysis curves for leached-unstabilized (LU) and leached-stabilized (LS) soil samples blended with 15% gypsum

that of the unstabilized ones. Table 4 represents the trend of the cohesion loss (Δc) of stabilized and unstabilized specimens containing 0%, 3%, 6%, 9%, 12% and 15% gypsum. Based on the table, increasing gypsum content of the stabilized and unstabilized samples increases the values of cohesion loss, significantly. For example, adding 12% gypsum to the pure soil increases the value of Δc by 0.38 and 0.53 kg/cm^2 of the stabilized and unstabilized samples, respectively. The increase in the cohesion loss (Δc) can be attributed to the presence of the gypsum in the soil texture and gypsum dissolution which weakens the inter-particle bonds between the soil aggregates and gypsum particles. It is observed that the stabilization of the soil samples with the nano-materials have a remarkable effect on preventing from the cohesion loss.

As shown in Fig. 10, Adding raw gypsum to the soil samples forms hexagonal CH gel which form several internal pores in the soil texture because of its specific shape. Adding Terrasil and Zycobond to the specimens fills the pores and enhances the cohesion as the most important factor controlling the shear strength of clayey soils. As an example, for the samples containing 15% raw gypsum, the addition of nano-chemical materials to the soil decreases Δc from 0.69 to 0.46 kg/cm^2, indicating that the improvement of the soil characteristics used in the study with the combination of Terrasil and Zycobond decreases the

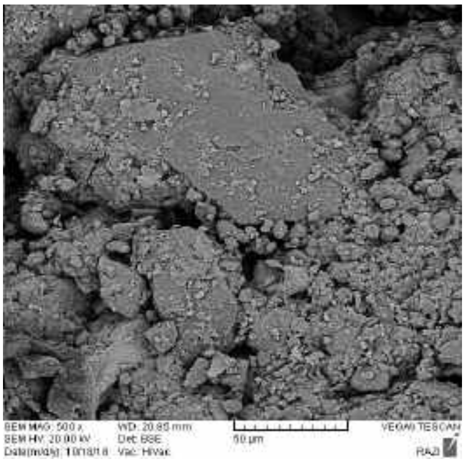

Fig. 13 SEM image of soil mixed with 3% raw gypsum (unstabilized sample)

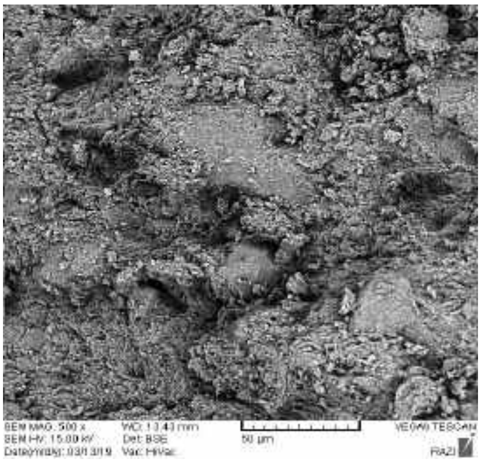

Fig. 14 SEM image of soil mixed with 3% raw gypsum (stabilized sample)

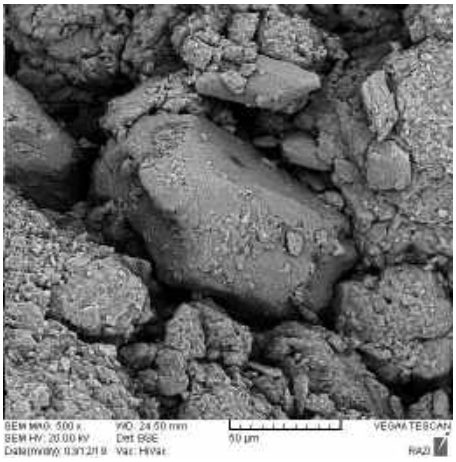

Fig. 15 SEM image of soil mixed with 15% gypsum (unstabilized sample)

cohesion loss of the sample by approximately 33%. The microstructural analysis including x-ray diffraction (XRD), scanning electron microscope (SEM) and the energy dispersive x-ray spectroscopy (EDS) were conducted on the unstabilized and stabilized samples remained from the modified consolidation tests. Figs. 11 and 12 provide the results of XRD analysis conducted on the samples containing 3% and 15% raw gypsum using Asenware AW-DX300 apparatus. By taking into account the XRD examinations and comparing the clay samples containing different percentages of gypsum, the researchers seek to find probable changes in the peaks of cementitious compounds such as calcium silicate hydrate (CSH) and calcium aluminate hydrated (CAH) which are one of the main factors that responsible for the increase in the shear strength of the stabilized soil samples in comparison with unstabilized ones. The changes in the intensity of the gypsum, sulfur, quartz, and illite peaks were investigated. The XRD graph of unstabilized and stabilized soil sample containing 3% raw gypsum indicates the presence of sulfur, gypsum, CSH, CAH and CH in the soil texture. The stabilization of the soil samples increases the intensity of gypsum peaks, indicating the dramatic effect of soil stabilization with Terrasil and Zycobond on preventing from gypsum leaching. This finding was proved using modified consolidation and EC tests. The XRD analysis of stabilized and unstabilized soil containing 15% gypsum, shown in Fig. 12, indicates the growth of gypsum, bassanite and CH peaks, along with the reduction of the intensity and fading of quartz, illite, CSH, and CAH peaks relative to the pure clay and the samples containing 3% gypsum. Therefore, the strength of the soil with 15% gypsum is significantly decreased compared to the pure clay and the soil with 3% gypsum, which is consistent with the results obtained from the direct shear tests. Despite the 15-days leaching, gypsum was found in the stabilized and unstabilized samples texture, indicating the long time process of gypsum dissolution.

The changes in the microstructure of stabilized and unstabilized soil samples containing 3% and 15% gypsum were evaluated using SEM technique. The chemical composition of the elements was also analyzed using EDS interpretation. The Tescan Vega-II XMU apparatus was used for this purpose and the samples were coated with gold of 100-angstrom thickness to avoid the charging phenomenon during imaging. The SEM images of the soil containing 3% raw gypsum in stabilized and unstabilized specimens, which are illustrated in Figs. 13 and 14, indicate the presence of internal pores.

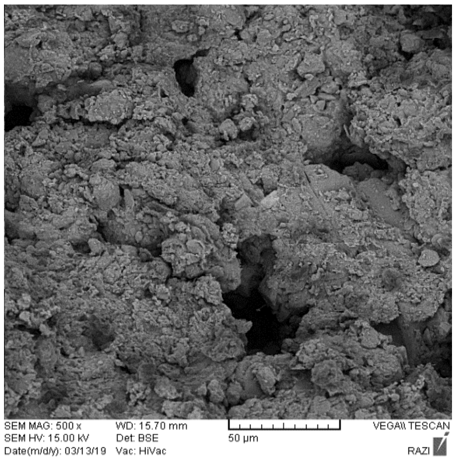

Fig. 16 SEM image of soil mixed with 15% gypsum (stabilized sample)

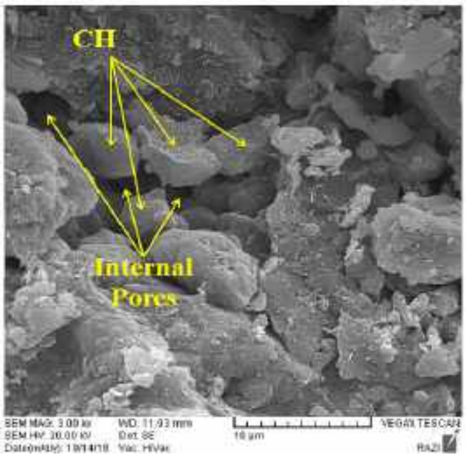

Fig. 17 Formation of several internal pores in presence of CH gel (unstabilized gypsifrous soil sample containing 3% gypsum)

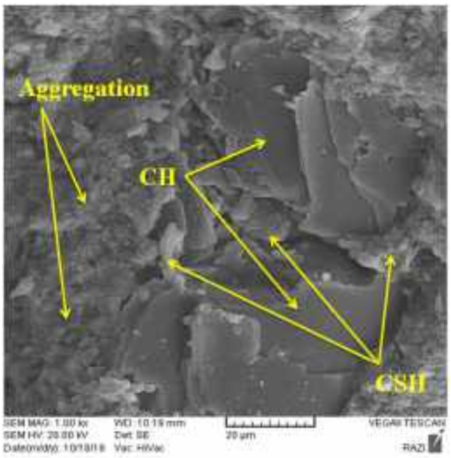

Fig. 18 Aggregation, Formation of CSH gel and a dense structure in presence of nano-materials (stabilized gypsifrous soil sample containing 3% gypsum

Table 5 Chemical composition analyses of soil-gypsum mixes for leached stabilized (LS) and leached unstabilized (LU) samples

Element/ Combination	Soil+ 3%G (LU)	Soil+ 3%G (LS)	Soil+ 15%G (LU)	Soil+ 15%G (LS)
Si	25.65	22.63	23.28	20.53
Al	4.15	3.89	3.22	2.95
S	0.73	0.87	2.98	3.66
Ca	1.04	1.35	6.02	6.61
Si/Al	6.18	5.82	7.23	6.96

The cementitious compounds such as calcium silicate hydrate (CSH) and calcium aluminate hydrated (CAH) were formed through improving the soil with nano- chemical stabilizer, and therefore, the soil texture was flocculated, which reduced the volume of internal pores and compressibility and densified the structure of the soil samples (Mirzababaei 2007, Roshni and Jeyapriya 2017). These phenomena can be attributed to considerable amount of calcium ion and sufficient time (15-days interval) for the reaction between ions of soil and gypsum.

Figs. 15 and 16 illustrate the SEM images of unstabilized and stabilized soil samples containing 15% raw gypsum, respectively. Due to the high rate of gypsum dissolution, the soil texture with 15% raw gypsum is looser and has larger volume of internal pores, compared to the similar samples with 3% raw gypsum. It is observed that the nano-chemical stabilizer used for soil improvement in the gypsiferous samples containing 3% raw gypsum performs better than that of the soil with 15% raw gypsum. The main reasons for this observation can be the weaker engineering properties of the gypsiferous soil samples containing 15% raw gypsum (due to remarkable amount of gypsum), the more leaching of the gypsum from the soil texture and consequently, the formation of more pores in the soil structure. These phenomena leads to an increase in the compressibility and a reduction in the strength of samples with 15% raw gypsum compared to those containing 3% raw gypsum which had been proved before by laboratory tests.

As shown in Fig. 17, adding raw gypsum to soil samples forms CH gel. Several internal pores formed due to hexagonal shape of CH gel which make the soil texture looser and weaken the engineering parameters of gypsiferous soil samples. Fig. 18 indicates that adding nano-chemical stabilizer to the soil specimens fill the internal pores that form in presence of CH gel. Also, aggregation and formation of cementitious compounds like CSH (calcium silicate hydrated) improve the engineering characteristics of the soil samples.

Table 5 reports the results of chemical composition analysis of the stabilized and unstabilized soil samples with 3% and 15% raw gypsum. Regarding the EDS spectrum, the silicon to aluminum ratio (Si/Al) in the soil with 3% raw gypsum is lower than that of similar samples with 15% raw gypsum, which is the reason for the lower strength of the lean clay samples with 15% gypsum compared to those containing 3% gypsum. It was also observed that for the samples with the same amount of gypsum, the stabilized ones have lower silicon to aluminum ratios (Si/Al) than that of unstabilized ones, proving their higher strength. In addition, an increase in the gypsum content in the soil from 3% to 15% significantly increases the amounts of calcium and sulfur in the stabilized and unstabilized samples, indicating the presence of more free gypsum in the soil which does not react with the soil particles, reduces the soil density and weakens the soil structure. This finding again highlights the justification of the considerable reduction in the soil strength by increasing gypsum content. By evaluating the effect of chemical stabilizer on the gypsum dissolution, the quantities of Ca and S in the samples with the same amounts of gypsum were higher in stabilized samples than those for unstabilized ones. This result implies that stabilization of the soil samples with Terrasil and Zycobond could reduce the gypsum dissolution from the soil texture, considerably. It should be noted that this result was concluded previously by conducting electrical conductivity (EC) test.

4. Conclusions

In this research, an experimental investigation was carried out to evaluate the effect of raw gypsum leaching on the engineering properties of a CL soil. Further, the combination of polymeric nanomaterials including Terrasil and Zycobond was used for stabilization of the lean clay samples containing various percentages of raw gypsum. The obtained results are as follows:

- By increasing applied stress and raw gypsum content in the lean clayey soil, the geotechnical parameters of the soil weakens, although the severity of weakness is higher and more destructive when the soil is subjected to a steady flow.
- The nano-chemical stabilizer used in the present study improves the engineering properties of the soil, due to its waterproofing characteristic and the formation of strong bonds between the particles in the clay samples containing raw gypsum.
- The waterproofing characteristic of the nano-material not only does prevent from the leaching of Terrasil and Zycobond in contact with the permanent and long-term flow of water, but also significantly reduces the gypsum dissolution, according to the results obtained from modified consolidation, electrical conductivity (EC), and direct shear tests.
- Regarding the waterproofing characteristic of the nano-material and the leaching period (15 days) in the laboratory tests, it can be concluded that the nano-chemical material used in the study can be used for practical long-term goals, such as the stabilization of embankments of water transmission canals.

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