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Experimental Investigation Of Mechanical Behavior Of Improved Marl Soil Using Nano Calcium Carbonate Ali Ohadian^a, Mehdi Mokhberi^{b,*}

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Abstract

One of the problematic soil types that is found in wide areas in the world is marl soils. This soil type is more sensitive to erosion and requires modifications in pavement construction processes. In this research, mixed soil-Nano-CaCo₃ was considered. For this purpose, 0.3 to 2.7% Nano-CaCo₃, by specific dry weight, was combined with marl soils in 0.3% increments. To analyse the geotechnical properties of marl soil, optimum water content for soil compaction, maximum dry density, uniaxial compressive strength, California Bearing Ratio (CBR) and the consolidation test were investigated. The results showed dry unit weight increases with increasing Nano-CaCo₃ content up to 2.2%. Furthermore, the CBR of modified marl soil with 2% Nano-CaCo₃ is 2.7 times greater than the initial CBR; it increased from 6 to 15.5 by adding up to 2% Nano-CaCo₃ was 2%, which increased the compression strength about 1.4 times. Besides, with increasing Nano-CaCo₃ up to 2.2%, the amount of void ratio decreases from 0.46 to 0.35. Additionally, the compression index C_c and swelling Index C_s are almost constant and equal to 0.09 and 0.03, respectively.

Keywords: Soil improvement, Nanomaterials, Nano-CaCo3, Marl soils, Problematic soils

1.Introduction

In constructing a building, the underlying soils transmit the imposed load to the subsurface layers. The soil must have sufficient strength to bear the applied pressures and should control the allowable settlement. Different soil types are known as problematic soils that have unusual mechanical and geotechnical behaviours, such as erosion, swelling, collapse and dispersion. Marl soil is a type of problematic soil that has swell, erosion and creep behaviour due to its constituents. This type of soil is widespread in many countries including the USA, UK, Iran, Iraq, Turkey, Azerbaijan and Saudi Arabia.

Marl soils are a rock or soil with 35–65% carbonate and corresponding clay content. It should be mentioned that this definition cannot be generally applied to many of the rocks that are often referred to as marls in the Persian Gulf area, Britain, the USA or other countries. For example, most of the marls of the Keuper series contain less than 20% carbonate material (Bell, 1996, Bell, 2013), while Ontario and Quebec marls are reported to have more than 79% calcite (Guillet, 1969.). The properties and behaviour of this type of soil have been considered by researchers. Such soils, according to the classification of clay–lime carbonate mixtures, are marl clays (Barth, 1939). Marl soils are very sensitive to erosion

and are one of the most important sources of sedimentation in catchments (Bouma and Imeson, 2000); (Jafari Ardekani et al., 2002). One of the inhibitor factors of the environmental behaviour of marl soils is its chemical properties (Sokouti and

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Razagi, 2015). Due to the great spread of marl rock in many countries, studying the characteristics of these formations is essential (Hooshmand et al., 2012). The emergence of various forms of erosion is one of the characteristics of marl areas (Ismail Nejad et al., 2007). Clay and silt are effective parameters in determining the sensitivity of marl soils derived soils to erosion (Sokouti and Razagi, 2015).

Modifying and improving bedrock and subgrades have a long history using mechanical or chemical methods. In a mechanical approach, the soils modify the texture of the soil and increase the mechanical strength of the soil by increasing the density and degree of internal friction. In chemical stabilization, the soil properties are modified by increasing the bonding between soil components. Therefore, soil improvement includes a wide range of materials such as lime, Portland cement, industrial slag, polymers, recycled materials, asphalt emulsion, etc. Among many stabilization materials, soil stabilization with lime or cement is the most widely used technique (Behnood, 2018, Sabat, 2012, Bell, 1996, Manikandan and Moganraj, 2014, Rao et al., 2011).

Nanotechnology is a relatively new approach in geotechnical engineering. Adding nanomaterial can modify the properties of soil, which implies the potential for its application in soil stabilization. The interesting features of these materials stem from the great number of atoms and molecules at their free surface and the resulting effects on their surface properties from physical, chemical and reactivity perspectives. Due to the high surface area and surface charge of nanoparticles, even small amounts of these additives can make noticeable improvements in soil behaviour. Nanotechnology has contributed to geotechnical engineering in two ways. First, it has facilitated the study of soil structure and the nature of its behaviour on a nanometre scale, and second, the analysis of soil behaviour on atomic and molecular scales have advanced the use of nanoparticles as soil

additives (Choobbasti et al., 2019, Meng et al., 2017, Changizi and Haddad, 2015)

A number of studies have been performed to investigate the behaviour of stabilized soil with nanomaterials [ex. (Choobbasti and Kutanaei, 2017, Meng et al., 2017, Tabarsa et al., 2018)]. (Majeed and Taha, 2013) reviewed a wide range of nanomaterials used in soil stabilization, as well as their effects. (Khalid et al., 2015) found that the compressive strength and effective shear strength were improved and the plasticity value decreased with a different number of nano-soil particles added to soft soil. The work of (Khajehzadeh et al., 2011) had a similar conclusion. The effect of different types of nanomaterial on the expansion and shrinkage behaviour of compacted residual soil was investigated by (Taha and Taha, 2012) and both swell and shrinkage strain were found to reduce with a certain proportion of nano-material.

Nano-CaCo₃ (NC) is a major nano-material currently being researched. (Liu et al., 2012) found that cement paste at an early age increased with the addition of Nano-CaCo₃ because the early hydration of cement was activated by Nano-CaCo₃ (Zhao, 2019). (Meng et al., 2017) investigated the effects of Nano-CaCo₃ on the properties of cemented soils in the marine environment. The addition of Nano-CaCo₃ improved the compressive strength, enhanced corrosion resistance and promoted hydration product generation.

(Sato and Beaudoin, 2011, Sato and Beaudoin, 2019) focused on the effect of Nano-CaCo₃ on the hydration of cement containing supplementary cementitious materials. Their results indicated the acceleration effect of early sample hydration was enhanced with more Nano-CaCo₃ added and the seeding effect of the Nano-CaCo₃ particles improved strength development. (Li et al., 2015, Supit and Shaikh, 2014) found that both Nano-CaCo₃ and Nano-SiO₂ (NS) could improve the compressive and flexural strength of concrete. NS acted as an effective filling material to reduce porous areas and accelerated the cement hydration process by a pozzolanic effect, and Nano-CaCo₃ acted mainly as an inert filler material to create a denser microstructure and accelerated the cement hydration process through a boundary nucleation growth effect.

Despite the problems with implementing projects on marl soils, insufficient studies have been carried out to

2. Materials and Method

2.1. Mechanical and Physical Properties of Soil

The marl soil was selected from the north-west area of Shiraz City. This type of soil is found in a wide area of southern Iran. This soil is a mixture of clay, calcium carbonate and gypsum. To evaluate the effect of

Table 1 Initial mechanical properties of studied marl soils

sustain these soils with modern stabilization materials. The main objective of this study was to investigate the effect of nanocarbons on the mechanical properties of marl soils. The parameters investigated in this study included Atterberg limits, maximum dry density, specific gravity, consolidation characteristics and California Bearing Ratio (CBR).

nanomaterials, the mechanical and physical properties of soil were measured using the same procedure proposed by ASTM standard codes. Totally, 8 group of tests performed to determine the initial specification of selected soils. Table (1) shows the mechanical properties of marl soil.

Optimum water content	Liquid limit (%)	Plastic limit (%)	Plasticity Index	Specific Gravity	Max dry density (kN/m ³)	Cohesive (C) (kPa)	Friction angle(φ) (Degree)
ASTM D2216	ASTM D4318	ASTM D4318	ASTM D4318	ASTM D854	ASTM D1557	ASTM D3080	ASTM D3080
13	32	23	9	2.7	18.7	100	21

2.2. Nano-Caco3

In this research, calcium carbonate nanoparticles, Nano-CaCo₃, with the characteristics presented in Table 3 were incorporated. The calcium carbonate type is used in many projects due to its highly reactive behaviour. The Nano-CaCo₃ additive was selected from US Research Nanomaterials Inc. Table (2) shows the proposed material properties.

2.3. Mineralogy And Microstructure

The mineralogy and microstructure of marlsoils was investigated using a combination of several techniques that include X-ray diffraction (XRD), X-ray fluorescence (XRF), and electron microscopy equipped with a scanning electron microscope, which can provide information on the chemical composition and microstructure of constituent particles, summarize the procedures and results obtained from these techniques, and the dominant mineralogical composition of marl soils.

Table 2

Chemical and mechanical characterization of Nano-CaCo₃ (US RN Inc.)

Colour	Brightness	Moisture	Specific Gravity g/cm3	DOP Oil Absorption ml/100g ≤	РН	MgO	SiO2	Al2O3	Fe2O3	Particle Shape	Activation Grade % ≥
White	96	<0.4	2-3	32	8- 10	<0.35	<0.1	<0.1	<0.1	Cubic	>98

2.3.1. Microscopic Illustration of Soils Structure

With the purpose of evaluating the materials used, the marl soils were photographed using a scanning electron microscope. Figures 1 shows the microscopy picture of a marl soils sample on the 30-micrometre



(a)

(b)





2.3.2. X-Ray Fluorescence (XRF)

The X-ray fluorescence (XRF) test is used for chemical analyses of rocks, minerals, sediments and fluids. This test has been used to determine the elemental composition of materials including metal composites, minerals, soils and rock. In this spectrometry method, the individual atoms, are excited by external energy and, therefore, obtain X-ray photons. XRF has the capability to analyse various elements, such as sodium, calcium, potassium and other minerals, in different concentrations from parts per million to high percentages. The results of the XRF analysis of the studied marl soil were carried out according to 92zr-4502 Standard. Table 3 shows the XRF analysis result of the studied marl soil.

scale. According to the picture, the examined soils

were composed of sand and silica and can be

considered as the boundary of sedimentation cycles.

Table 3

Elemental analysis of Nano-CaCo3 used in research from XRF decomposition

Compound	
	marl
SiO ₂	26
CaO	27
Al ₂ O ₃	8
MgO	6
Fe ₂ O ₃	5
K ₂ O	2
TiO ₂	0.5
Na ₂ O	0.4
SO ₃	0.2
LOI*	24.9
Loss on Igni	tion (1000°C, 2 h)

2.4.Tests Procedures

To evaluate the effect of Nano-CaCo₃ on the geotechnical parameters of marl soils, different dry weight percentages of Nano-CaCo₃ were separately added to the soils. The main purpose of this research was to determine the percentage of Nano-CaCo₃ and soil mixture that exhibits the highest strength and the least deformation of underlying soils. Since a disproportionate distribution of nano in the soil reduces the efficiency of nano-stabilized soils, it is necessary to mix the soil and Nano-CaCo₃ in a suitable manner to achieve a homogeneous composition. Various methods, such as ball milling, have been developed to evenly distribute these particles in the

soil. In this regard, the dry soil samples, mixed with different percentages of Nano-CaCo₃ were poured into a ball mill for one to two hours. Each mixture was stirred slowly while adding an accurate amount of water, to achieve a homogenous stabilized soil mixture with a water content. To ensure a more uniform moisture distribution throughout the mixed soil-nano-CaCo₃-water were stored in a sealed container for 16 h. Modified marl soils were tested for the maximum dry density, optimum water content, unconfined compressive strength, consolidation and California Bearing Ratio (CBR) using the appropriate ASTM standards. Totally, 56 different tests carried out to

evaluate the effect of Nano-CaCo₃ on mechanical properties of marl. the tests results are summarized in

3.Results and Discussion 3.1.Standard Proctor Compaction Tests

The purpose of the compaction test is to determine the maximum dry density of the soil at optimum water content. The standard proctor test was performed for both the natural soils and the soil-Nano-CaCo₃ mixtures to determine the compaction parameters. The specimens were prepared following the ASTM D697-07 standard as a base for these tests. The soils were sealed in plastic bags and allowed to hydrate for at least 24 h before compaction. The standard proctor compaction curves were found to be significantly affected by the addition of small amounts of Nano-CaCo₃ (i.e., between 0.3% and 2.7%), as shown in

3.2.Compressive Strength Unconfined Test

The uniaxial shear strength (UCS) of adhesive soil is measuring by the unconfined uniaxial test. This test used to determine in-place soil strength. The test is determined as a triaxial shear test with zero lateral compressive stress. In fact, the shear strength of soil depends on friction angle and coefficient of adhesion. The friction shear strength is the internal force commonly seen in granular soils, whereas the adhesive shear strength is a function of surface forces. Due to its facility and simplicity, the unconfined compressive strain control test was preferred over the stress control test. The soil strain ranged from 0.5 to 2% per minute. Samples for this test were prepared from modified marl soils.

To achieve maximum strength, the samples were prepared with the calculated optimal moisture in the

2.2.4. California Bearing Ratio (CBR) Test

The CBR test has been used to determine the bearing capacity of the subgrade, sub-base and base of pavement, and roadbed. This is the generally used method for determining the thickness of flexible pavement. This test was performed according to table (4). The data were thoroughly analysed and the results are shown in the respective graphs.

Figure 2 and Figure 3. There was a consistent and fairly significant increase in the dry unit weight with increasing Nano-Caco3 content, with the maximum dry unit weight decreasing from 19.1 to 19.8 kN/m3 as the Nano-Caco3 content was increased from 0% to 2.2%. The optimum water contents increase by about 3.7% with the addition of 2.5% Nano-CaCo₃, after which it decreased in a consistent manner by about 1.3% as the Nano-CaCo₃ content was increased from 2.2 to 3%. The effect of Nano-CaCo₃ on the shape of the compaction curve is slightly more pronounced on the dry side of optimum.

compaction test. Calculations were based on axial stress amount and strain ratio, and the ultimate stress value was obtained from stress-strain curves. If there was no definite maximum strength, the strength at 11 to 15% strain was considered the maximum and the slope at the origin of the stress-strain curve was calculated as the initial elasticity modulus.

As Figure 4 shows, adding Nano-CaCo₃ to marl soil increased the uniaxial compressive stress. The optimum percentage of Nano-CaCo₃ is 2% which increased the compression strength about 1.4 times. The compression strength is decreased by adding more Nano-CaCo₃ than 2%.

ASTM D1883-87. The CBR test was carried out on compacted samples at an optimum water content. To perform the CBR test, the samples were prepared according to the modified compaction test (ASTM D1557). Marl soils and various percentages of Nano-Caco3 were mixed. The compaction test was performed at certain intervals to achieve an optimum

moisture percentage. The soil-Nano-CaCo₃ mixture with the optimum water content, as determined in the compaction test, was subjected to the CBR test.

Table 4	
Geotechnical properties of Nano-CaCo3 improved ma	rl soils

Test		Percent of mixed Nano-CaCo ₃ (%)									
Commention	Optimum water content (%)	13.5	14.2	14.9	15.3	15.8	16.6	17.1	17.7	17	16.4
Compaction	$(\gamma_d)_{max}$ (kg/cm ²)	1.92	1.93	1.935	1.93	1.94	1.94	1.96	1.98	1.96	1.93
UCS (kN/m ²)		197	202	213	234	242	252	260	274	253	212
Consolidation	Cc	0.13	-	-	0.15	-	-	0.1	-	-	0.098
	Cs	0.05	-	-	0.033	-	-	0.044	-	-	0.054
	e_0	0.46	-	-	0.39	-	-	0.35	-	-	0.4
CBR		6	8	9	9.5	10.7	12	14	15.5	12.3	7.7



Fig.2.Variation of optimum water content marl soil modified with Nano-CaCo3



Fig.3. Variation of maximum dry density of marl soil modified with Nano-CaCo3

According to the results, the CBR values of soil samples modified with Nano-Caco3 increased relative to the initial soil sample as well as during the curing time. The initial CBR was 6. Based on Figure 5, the CBR is increased up to 2%. The CBR of modified soil with 2% Nano-CaCo₃ was 2.7 times the initial CBR

and the soil had a tendency toward coarse granulation in terms of soil classification. As a result of hydration in the Nano-CaCo₃-soil mixture, soil particles get together and stabilize, and soil strength parameters increase.



Fig.4. Variation of unconfined compression strength of marl soil modified with Nano-CaCo3



Fig.5.Variation of CBR of marl soil modified with Nano-CaCo3



Fig. 6.Variation of resilient modulus of a subgrade with the CBR of marl soils

According to Equation (1), for resilient modulus (M_r) in soils based on the AASHTO Soil Classification System:

$$M_r(\frac{kN}{m^2}) = 18000 \times (CBR)^{0.64}$$
(1)

In an applied perspective, the increase in the resilient modulus is associated with an increase in the layer

2.2.5. Consolidation Test

To investigate the consolidation characteristics of marl soil mixed with nano-CaCo₃, three Nano-CaCo₃ contents (0.9, 1.8 and 2.7%,) were selected and compared with the results of pure marl soil. The specimens were prepared using the same procedure described in the previous sections.

The one-dimension consolidation test was performed following the ASTM D2435 procedure recommended for an oedometer test. First, the initial void ratio of soil samples was measured, as presented in Table 4. After that, the soil samples were consolidated with different percentages of nano-materials. This process continued coefficient; as a result, the thickness of the designed pavement layers decreased. This coefficient was 1.6 times the initial value in the soil sample modified with 2% Nano-CaCo₃ by weight. Figure 6 illustrates the relationship between the resilient coefficient of marl soils and the amount of CBR.

until a stress of 8 kg/cm² was reached. Last, an unloading step was performed by decreasing the load to 2 kg/cm^2 .

As can be seen, with increasing amounts of Nano-CaCo₃, the void ratio decreases due to the fine pores filling with nano-material. This procedure continues until the Nano-CaCo₃ ratio reaches the optimum amount; after that, the void ratio is increasing with increasing Nano-CaCo₃ amount. Figure 7 shows the void ratio changes with the addition of Nano-CaCo₃ in marl soils.



Fig.7.Variation of the initial void ratio by variation in Nano-Caco3 per cent



Fig.8. e-log curve of the consolidation test for Nano-CaCo3 modified marl soils

Figure 8 shows the e-log(p) curves to illustrate the consolidation behaviour of improved marl soils. The slope of the loading and unloading graphs, which are equivalent to the compression index (Cc) and the swelling index (Cs), were calculated. According to the figures, Cc and Cs are almost constant and equal to

3.Conclusions

In this research, the effects of Nano-CaCo₃ on the geotechnical properties of marl soils were investigated. The samples were examined for

0.09 and 0.03, respectively, unless their void ratios are varied by Nano-CaCo₃ content variation, as discussed above.

Due to the wide distribution of marl soil in the world, it is recommended that improving of marl soil evaluated with other types of nanomaterials.

Atterberg limits, compaction, unconfined compressive strength, CBR and consolidation. The following

conclusions were drawn from analysing the test results:

- 1. There was a consistent and fairly significant decrease in the dry unit weight with increasing Nano-CaCo₃ content as the Nano-CaCo₃ content increased from 0 to 3%.
- 2. The optimum water content decreased by about 0.6% with the addition of 0.5% Nano-CaCo₃, after which it increased in a consistent fashion by about 1.3% as the Nano-CaCo₃ content was increased from 0.5% to 3%.
- 3. According to the results of unconfined compressive tests, the best improvement in sample strength, which was about 100%, was achieved by adding 2% Nano-CaCo₃. This is due to the surface area of Nano-CaCo₃, which increases surface area by decreasing the particles size and, therefore, caused an increased surface area reaction between the Nano-CaCo₃ and soil particles. An increase of 40% in unconfined compressive strength is observed with 2% Nano-CaCo₃.
- 4. According to the results, by adding small amounts of Nano-CaCo₃, the CBR values increased in the soil samples. The CBR of

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modified soil with 2% Nano-Caco₃ was 2.7 times the initial CBR. Moreover, adding up to 2% Nano-CaCo₃ to marl soils increases the resilient modulus by 1.5 times compared to the initial CBR.

- 5. Mixing 0.3% to 2.7% Nano-CaCo₃ into the marl soil has little effect on the original consolidation characteristics. However, by increasing the pressure, the final settlement is gradually increasing. It seems that Nano-CaCo₃ have less effect on the compression index and swelling Index, and these parameters are almost constant unless the void ratios are varied by Nano-CaCo₃ content variation.
- 6. After adding a small amount of Nano-CaCo₃, the marl soil exhibits different consolidation characteristics. When the Nano-CaCo₃ mixing content is 1.8%, the final settlement reaches a maximum and the final void ratio is the smallest. Under high-pressure conditions, the consolidation increases with the increase in Nano-CaCo₃ mixing content, which indicates that Nano-CaCo₃ modifies the consolidation of marl soils.
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