

Stabilizing clay soils incorporating nano materials

(through a sustainable development approach)

Abolfazl Soltani^{1*} Amin Moradi² Nabi-allah Ahmadi³

^{1*}Corresponding Author, Associate Professor in Geotechnical Engineering, School of Civil Eng., Shahid Rajaei Teacher Training University (SRTTU), Tehran- Iran. Email: asoltani@sru.ac.ir

²Master in Geotechnical Engineering SRTTU, 1678815811, Tehran, Iran. a.moradi@sru.ac.ir

³PhD. Student in Geotechnical Engineering, SRTTU, 1678815811, Tehran, Iran. ahmadi@sru.ac.ir

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Abstract

In this paper, the performance of some nano materials including multi wall carbon nano tubes (MWCNTs), carbon nano fibers (CNFs), nano silica, nano kaolinite and nano montmorillonite is compared in order to evaluate geotechnical properties and elastic modulus of the stabilized clayey soils. The mechanical tests were carried out on samples treated by nano particles indicate that MWCNTs and CNFs are essential additives for improving the physical properties of unstable soils including Atterberg limits, optimum moisture content and maximum dry density. Small amounts of nano silica and nano kaolinite particles increased the maximum dry density, the unconfined compressive strength (UCS) and the elastic modulus (E) results. However, a reverse trending in optimum moisture content is observed in which nano silica treated the soil towards the higher water contents. In contrast, nano kaolinite performs a higher positive influence on the stiffness of the treated specimens using a significant amount of less water absorption. It is concluded that the major difference between the performances of all the studied nano particles includes that nano montmorillonite reduced the dry density of soil as the $\omega\%$ increased. This type of soil stabilizing is in agreement with sustainable development strategies.

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INTRODUCTION

Improving the geotechnical properties of soil with nano materials is a new effective method for stabilizing the unstable clay soils. There are various types of natural and industrial nano materials which are known as soil-additives in geotechnical proposes. Nano materials are different in microstructure, crystal shape and chemical capability, causing variation in performances of the soils to be stabilized. This method is applicable, economic and durable in the reduction of collapse, failure and project costs. In past decades, the process of soil stabilization was carried out with traditional cementitious and reinforcement materials such as: cement and lime particles or straw fields (Basha et al., 2005; Jauberthie et al., 2010; Ghobadi et al., 2014; Khemissa and Mahamedi, 2014). However, improving the geotechnical properties of soil by cement and lime in the presence of water depends on their

chemical reactions with the soil elements (Azadegan et al., 2012; Khemissa and Mahamedi, 2014). Other additives, such as: straw fields and rice husks to improve soil properties, depend on their physical effects (Lu et al., 2014). There are main disadvantages to these chemical stabilizers including global atmospheric pollution by CO₂, increased swelling potential of soils and high energy consumption (Ramaji, 2012; Sadeghi-Nik et al., 2017). In recent decades, the production of active particles with different approaches such as: pozzolanic and nano materials have been proposed for soil stabilization (Manso et al., 2013; Alujas et al., 2015; Correia and Rasteiro, 2016). Preparation of nano materials includes the extraction of desired nanoparticles from natural, industrial and organic materials using various techniques, including energetic stirring followed by centrifugation and freeze drying, sonication and filtration, grinding with ball mill instrument (Franco et al., 2003; Khalid et al., 2005; Zsirka et al., 2015). The preparation of nano materials depends on their types of application and the way the soil performs including physical, chemical and physio-chemical processes. The present work attempts to distinguish and compare the geotechnical performance and evaluate mechanical properties (e.g. elastic modulus) of stabilized clay soils by some nano materials like multi wall carbon nano tubes (MWCNTs), carbon nano fibers (CNFs), nano silica, nano kaolinite and nano montmorillonite.

Methods and Equipment

New methods have been proposed to observe the micro structure of clay soils at nano metric scales including scanning electron microscope (SEM), transmission electron microscope (TEM) and atomic force microscope (AFM). These are direct methods for taking images of nanoscale particles and they provide information concerning the dimension, shape, and morphology of nano materials. SEM was invented in 1931 by Knoll and Ruska. It can provide larger images using backscattered electron. In this method, an electron ray perpendicularly shines on the specimen. As an interaction between the ray and specimen in vacuum state, the electrons and X-rays are emitted. Sensors receive the emitted X-rays, the initial electrons, and the resulting electrons from the interactions of the initial electrons (Hawkes, 2004). The sensors convert them into signals and transmit signals to monitors which provide the final image. This method can be also used to investigate the size and distribution of the nanoparticles.

The exact particle size of nano kaolinite samples, the mineral morphology and microstructure of the kaolin samples for this study, were obtained by (FE-SEM), TESCAN-MIRA III model, a completely new field emission scanning electron microscope. This instrument unit is made in the Czech Republic, available in Beam Gostar Taban Laboratories, Tehran, Iran. To prevent electron surface charging during FE-SEM, samples were gold coated.

TEM was invented in 1939 by two German scientists (Siemens and Halska) to simplify the analysis process and improve the accuracy of images. Like SEM, TEM uses electron emissions by passing the electrons through the specimen to a phosphorus detector to provide a plan of the specimen structure. TEM is able to take images of the produced nanoscale particles so that, the size and morphology of particles can be scanned. It also can be used to differentiate the presence of inorganic materials of soil. In addition, it enables the

identification and analysis of features at significant sub-micrometer scales (Williams and Carter, 1996; Zhu and Lu, 2010).

AFM was invented in 1986 by Binnig Quate, and Gerber. It has many applications in determining the surface topography and studying the surface cavities. AFM consists of a cantilever with a sharp tip at its end that is used to scan the specimen surface. AFM unlike an electron microscope device which provides a two-dimensional projection or a two-dimensional image of a sample, provides a true three-dimensional profile of the surface (Lang et al., 2004). It is noticeable to realize that the high resolution of AFM is comparable to SEM as well as TEM.

The AFM image for nano kaolinite is used in geotechnical engineering to study the roughness of surfaces and the friction angle between soil particles. A major advantage of AFM is that, unlike electron microscopy, it can operate in air, vacuum, and liquid environments. In this study, the microstructure of nano kaolinite was observed by an ARA AFM instrument in contact mode at Shahid Rajaee Teacher Training University, Tehran, Iran. The imaging process was performed at room temperature using a silicon cantilever with a nominal spring constant of 40 Nm^{-1} and a resonance frequency of 325 kHz, equipped with an ultra-sharp silicon tip. The AFM, SEM and TEM images of this study are shown in Figures 1-3, respectively.

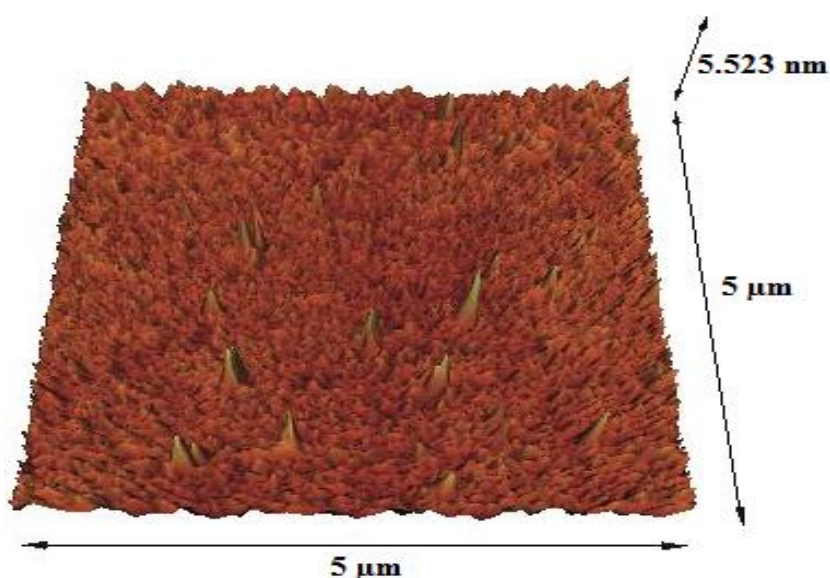


Figure 1. AFM (Atomic Force Microscopic) image for processed nano-kaolinite was prepared after final ultrasonic dispersing. The image shows a geometrical configuration of ultra-fine nano-kaolinite particle sizes and very little surface roughness. It also shows that except for a few peaks, other particles have a height of less than 5 nm, the finest possible size of nano particles for soil studies in geotechnics.

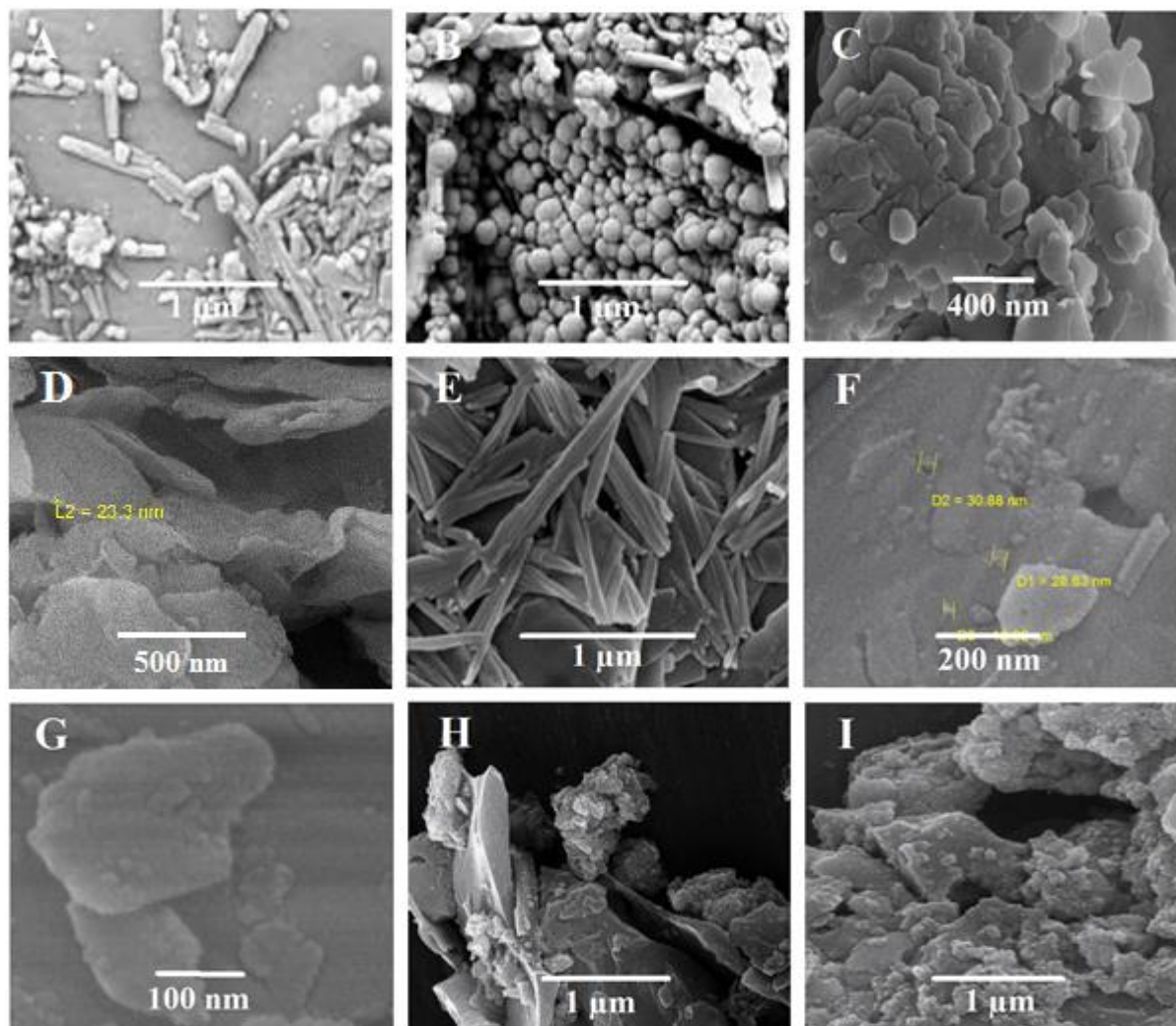


Figure 2. SEM images showing microstructure and morphology of some common clay minerals including: nano-tubes halloysite (A), nano-spheroidal halloysite (B), nano-tabular halloysite (C) ([Lázaro, 2015](#)), nano montmorillonite (D) ([Sadeghi-Nik et al., 2017](#)), nano-scrolls kaolinite (E) ([Liu et al., 2016](#)), nano-kaolinite flakes (F, G, H) and cabbage growth kaolinite (I).

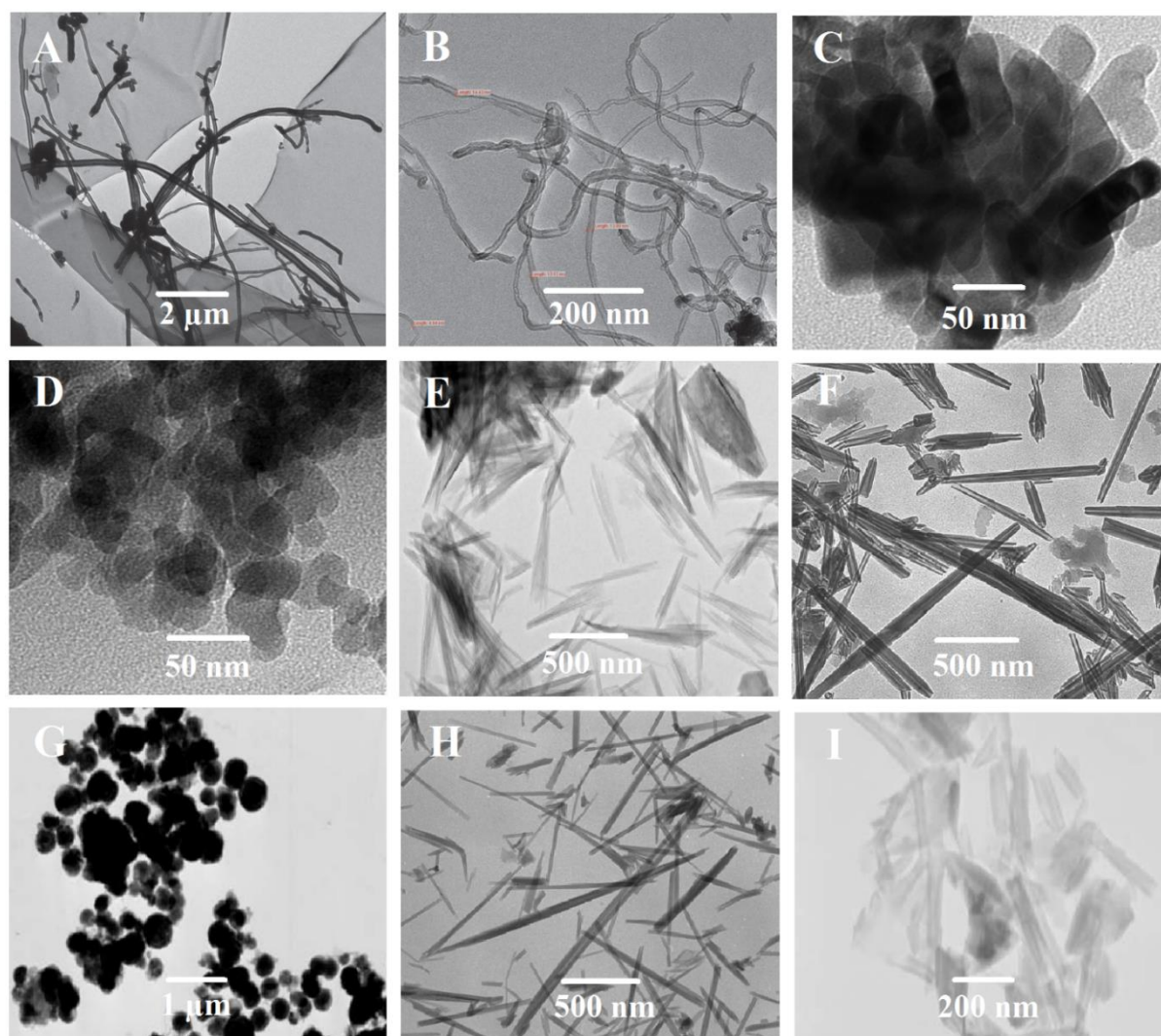


Figure 3. TEM images showing morphologies of multiwall carbon nanotube (A), carbon nanofiber (B) (Alsharef et al., 2016), nano TiO_2 (C), nano SiO_2 (D) (Xu et al., 2018), nano-scrolls kaolinite (E) (Liu et al., 2016), halloysite nano-tubes (F), halloysite nano-spheroidal (G) and halloysite long needle-shaped tubes (H and I) (Lázaro, 2015).

Effects of nano materials

Multiwall Carbon Nanotubes and carbon nano fibers

Major and minor advantages of nano particle additives into various soils are presented in Table 1. Among them, five common nano materials were selected to verify the geotechnical consideration by this study. Atterberg limits results for samples containing MWCNTs (Figure 4A) and CNFs (Figure 4B) indicate that by increasing carbon nanomaterials, no significant change is observed. A slight increase in plastic limit (LL) and plasticity index (PI) is observed only when 0.05 to 0.1 wt% of nanomaterial is added, and then encompasses through the constant trends. This means that carbon nanomaterials increase the water absorption capacity of soils due to large surface area. Also, the PL value for soil 1 is almost constant except for incorporation of 0.075 wt% of MWCNTs where PL slightly decreases (Figure 4A) and for 0.05 wt% of CNFs in which slight increase is observed (Figure 4B). The lack of a substantial trend for variation in Atterberg limits and PI means that carbon nanomaterials may contribute and were dispersed physically rather than to enter in

chemical reactions. To identify the performance of unique proportions of carbon nano-materials mixed with control sample (Figures 5A, 5B), dry density and moisture contents were precisely determined. The above figures show that due to the scattering results, no specific trend is observed for compaction characteristics. The slight decrease in moisture content (shown in Figures 5A and 5B) is related to the tendency of the nanocarbons to fill pores between the control sample particles. In Figure 5A, containing control sample information reinforced by MWCNTs, it is observed that the maximum decrease in optimum moisture content is 13.5% at a dose of 0.075% MWCNTs, and dry density increases from 1860 to 1890 kg/m³. For soil sample reinforced by CNFs (Figure 5B), the maximum decrease in optimum moisture content is 14% at dose of 0.075% CNFs. Furthermore, dry density increase from 1860 to 1880 kg/m³ (e.g. Taha and Alsharef, 2018).

Nanosilica

The relationship between liquid limit (LL) and nanosilica content of stabilized control sample is illustrated in Figure 4C. From Figure 4C, one can be concluded that adding 1 wt% nanosilica leads to a reduction of LL from 56.4 to 47.8%, and by adding proportions of nanosilica, the PI decreases by around 52% compared to the natural clay (control sample). The above figure also indicates that an increase in nanosilica content leads to an increase in the PL of stabilized control sample. Compared to natural clay (control sample), the plastic limit is increased to 6, 13 and 15% as the corresponding values of nanosilica is increased from 0.5, 0.7 to 1%. A further increase in percentage of additive beyond optimum for soil stabilization causes a reduction in overall rate of PI of the stabilized soil (Changizi and Haddad, 2017).

It is worthwhile to mention that an increase in nanosilica content contributes to an ideal increase in the maximum dry density and optimum moisture content of soil stabilized with nanosilica (Changizi and Haddad, 2017). A range of maximum dry density varying from 1640 to 1820 kg/m³ was obtained depending on the nanosilica proportions for control sample (Figure 5C). This variation is nearly equivalent to the variation of moisture content from 15 to 18%. The results of the unconfined compressive strength (UCS) tests on treated samples are presented in Figure 6A. As the additive proportions increase, the USC peak of the stabilized clays (control sample) increases (Figure 6A). Thus, the optimum nanosilica is 0.7 wt%. By adding nanosilica, the increase ratios of UCS include 1.3, 1.56 and 1.5 for the corresponding nanosilica proportions of 0.5, 0.7 and 1.0 wt%, respectively (Figure 6A).

Nanokaolinite

To identify the performance of nanokaolinite, the control sample was mixed with 1, 3 and 5% of nanokaolinite portions (Figure 4D), and it showed a remarkable increasing trends from 25.96 to 32.68% and 16.14 to 18.74% for LL and PL values, respectively. Due to higher intensity increas of LL compared to PL, the PI percentages also increased from 9.82 to 13.94%. Another important interesting performance in compaction characteristics is that in the control sample which was treated by different doses of nanokaolinite, dry density increased from 1902 to 1951 kg/m³ and the optimum moisture content reduced from 12.44 to 10.09% (Figure 5D). The UCS values (Figure 6B) show that improvement of control sample by nanokaolinite starts at >1 wt% and continues to higher strengths by up to 5 wt%, compared to the control sample. By adding nanokaolinite to control sample, the increased

ratios of UCS are 0.73, 2.13 and 3.60 corresponding to 1, 3 and 5 wt% of nanokaolinite, respectively.

Nano montmorillonite

According to Figure 4E the Atterberg limits including LL, PL and PI increase by increasing the nano montmorillonite contents. However, a reverse trending is observed between dry density and moisture content (Figure 5E), in which by increasing the portions of nano montmorillonite i.e. from 0.5% to 3%, the maximum dry density reduces from 1520 to 1343 kg/m³. The optimum moisture content decreased by about 0.6% when adding 0.5% nano montmorillonite, after that, this trend consistently by about 1.3% as the nano montmorillonite content was increased from 0.5% to 3%. The effect of nano montmorillonite on the shape of compaction curve is more pronounced on the dry side of optimum. As shown in Figure 6C, there is a significant increase in UCS of the control sample with 2% nano montmorillonite specimens coinciding with increases in the nano montmorillonite content, shows a two-fold increase in strength compared to the strength given by control sample. This behavior may be resulted from high ion exchange coefficient of nano montmorillonite as evidenced from high MgO and CaO contents in chemical composition (Sadeghi-Nik et al., 2017; Tabarsa et al., 2018).

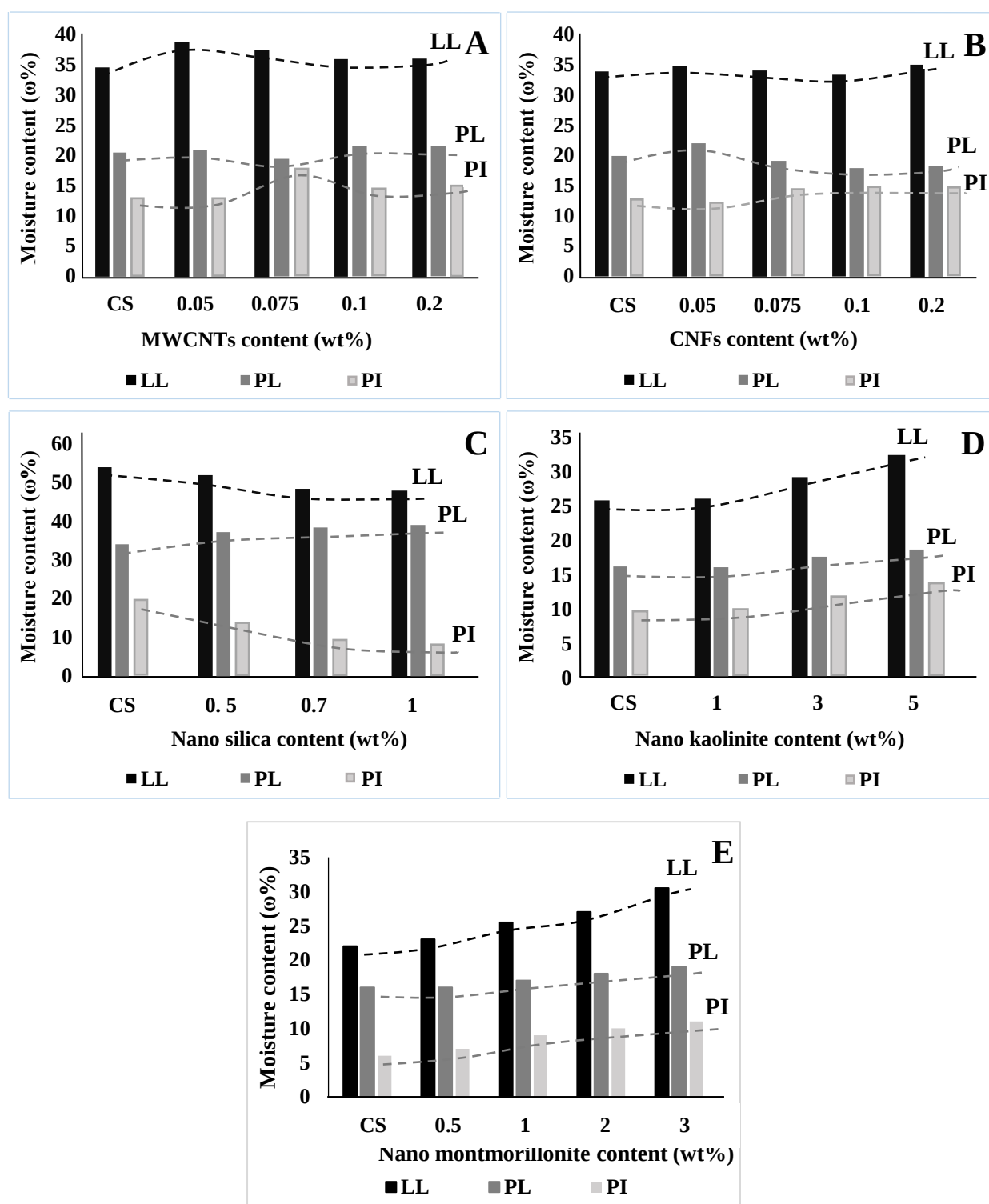


Figure 4. The Atterberg limits for specimens stabilized by MWCNTs (4A), CNFs (4B) (Alsharef et al., 2016), nano silica (4C) (Changizi and Haddad, 2017) nano kaolinite (4D) and nano montmorillonite (4E) (Tabarsa et al., 2018). CS = control sample; LL = liquid limit; PL = plastic limit; PI = plasticity index.

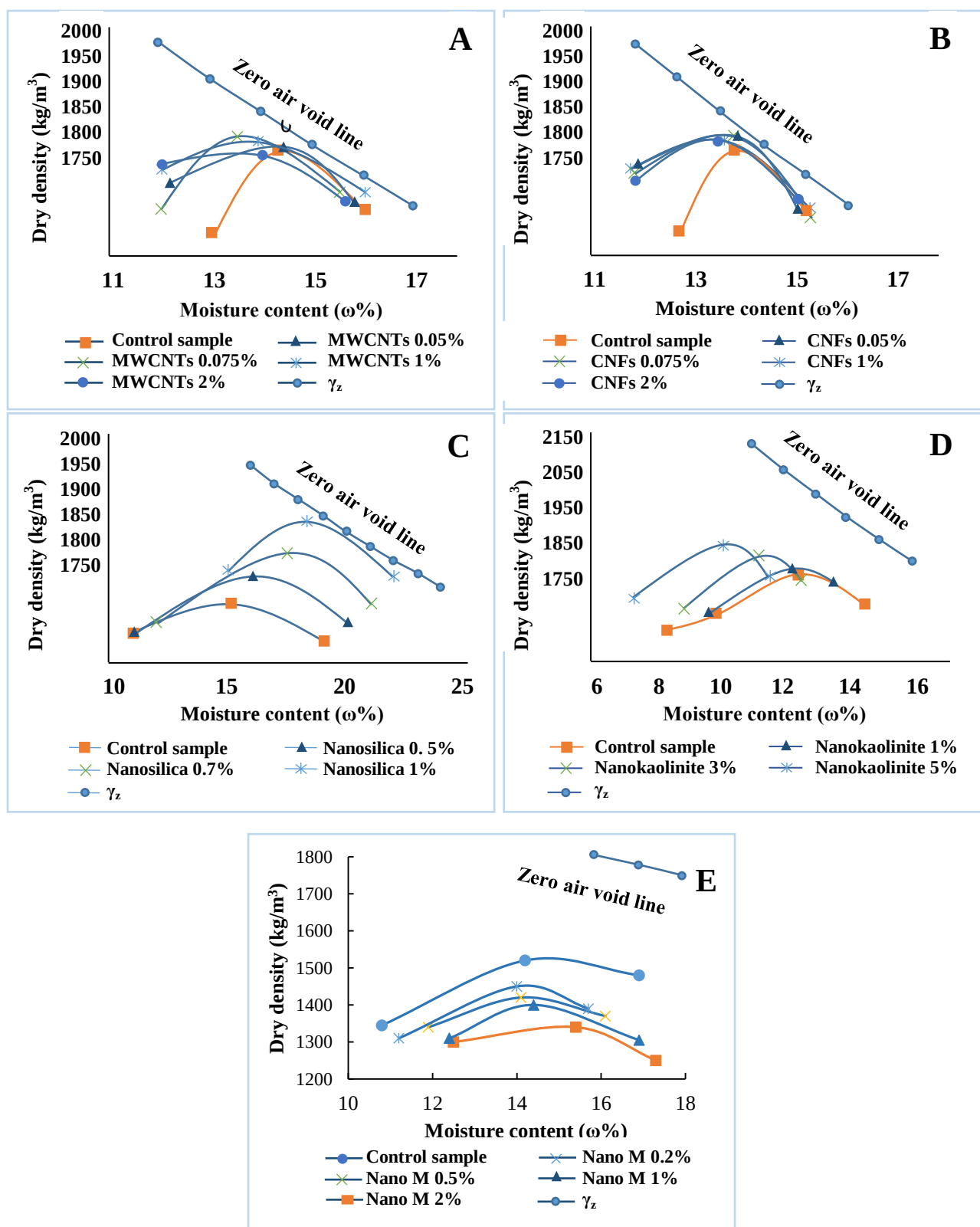


Figure 5. Compaction curves for specimens stabilized with MWCNTs (5A), CNFs (5B) (Alsharef et al., 2016), nano silica (5C) (Changizi and Haddad, 2017), nano-kaolinite (5D) and nano montmorillonite (Nano M) (5E) (Tabarsa et al., 2018).

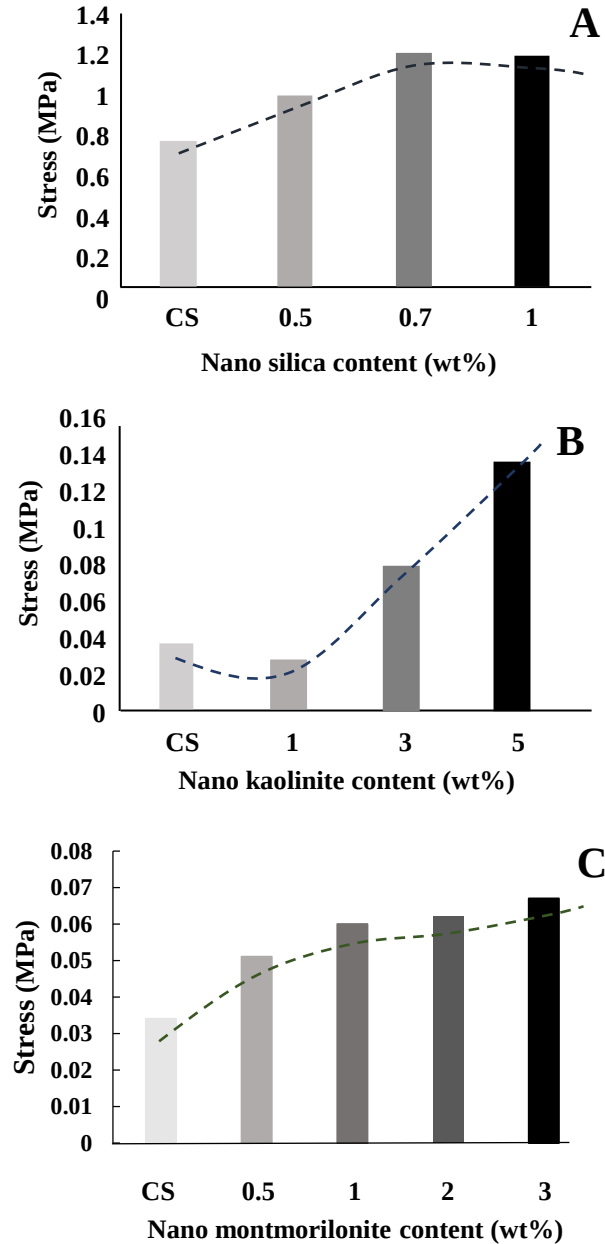


Figure 6. The UCS test results for soil stabilized with nano silica (6A) (Changizi and Haddad, 2017), nano kaolinite (6B) used in current study and nano montmorillonite (6C) (Tabarsa et al., 2018); CS = control sample.

Elastic modulus

Major development in UCS tests related to MWCNTs and CNFs is not observed. This behavior is merely assumed to be the result of physical performance of carbon nano materials rather than chemical reactions. Therefore, the modulus of elasticity (E) for carbon nano materials is not fully specified. However, the elastic modulus data obtained from UCS results for clay stabilized with other nano particles is appreciably observed in Figure 7. This figure shows that, by increasing

in nano silica content (Figure 7A), the increased elastic modulus ratios are 1.2, 1.41 and 1.84, respectively for nano silica contents of 0.5, 0.7 and 1 wt%. The calculated corresponding values for nano kaolinite indicates that by increasing nano kaolinite from 3 to 5 wt%, the elastic modulus ratios increase from 2.45 to 3.8 (Figure 7B). Therefore, nano kaolinite can enhance the elastic modulus of clay soil better than nano silica contents. This characteristic may be related to the method of nano preparation, the higher degree of nano kaolinite structural disorder and/or the different dimensions in the crystallographic directions of nano kaolinite (Franco et al., 2004). It should be noted that the progress in modulus of elasticity for nano montmorillonite is not significant when the mix proportions increase from 0.2 to 2 wt%.

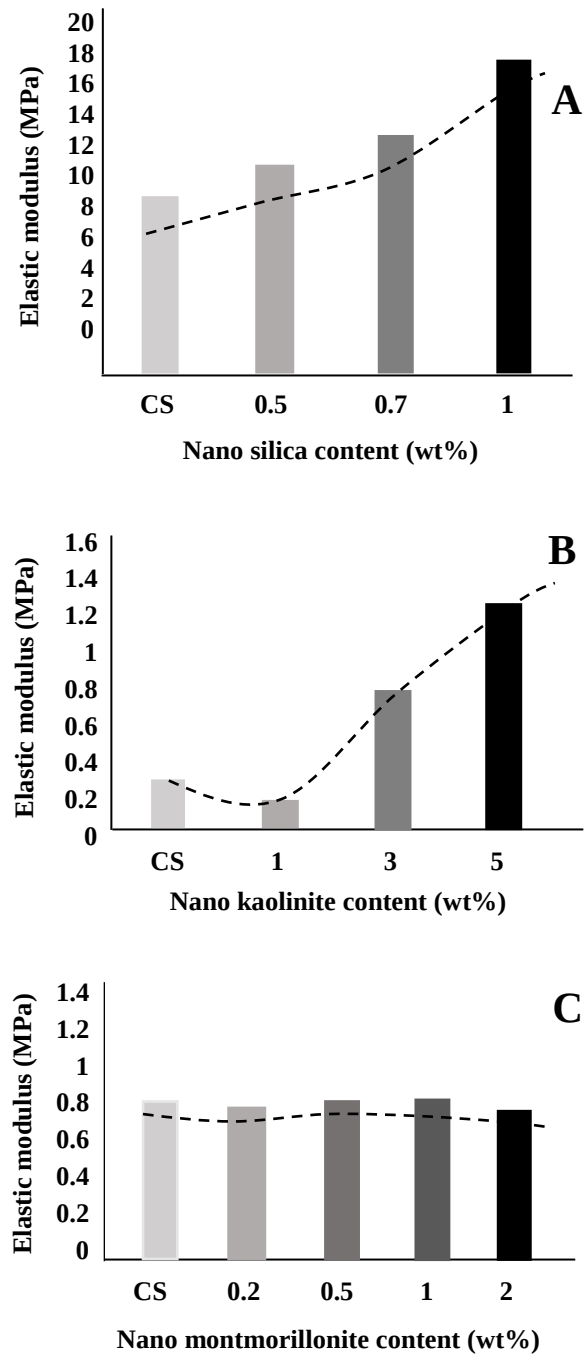


Figure 7. The elastic modulus results for soil stabilized with nano silica (7A) (Changizi and Haddad, 2017), nano kaolinite (7B) used in current study and nano montmorillonite (7C) (Tabarsa et al., 2018); CS = control sample.

Table 1. Major and minor advantages of nanoparticle additives into different soils.

Type of soil	Nanoparticles/additives	Proportions	Conducted tests	Advantages		References
				Major	Minor	
Silt	Multiwall carbon nanotubes and cement	0.001, 0.01 and 0.1 (g)	UCS	Improved UCS	-	(Correia et al., 2015)
Clay	Nanosilica and cement	0.2, 0.4, 0.8 and 1 wt%	Atterberg limits, Proctor compaction, UCS, Permeability	PI, optimum $\omega\%$ and UCS increased.	Hydraulic conductivity decreased.	(Bahmani et al., 2014)
Sandy clay	Multiwall carbon nanotubes and carbon nanofibers	0.05, 0.075, 0.1, and 0.2 wt%	Atterberg limits, Proctor compaction, Hydraulic conductivity	LL, PI, optimum $\omega\%$ and dry density increased.	PL and Hydraulic conductivity decreased.	(Alsharef et al., 2016)
Clay	Nanosilica and lime	5, 10 and 15 wt%	Atterberg limits, Proctor compaction and UCS	Optimum $\omega\%$ and UCS increased.	LL, PI and maximum dry density decreased.	(Kirithika et al., 2015)
Loess	Nanoclay	0.5, 1, 2 and 3 wt%	Atterberg limits, Proctor compaction, UCS, UU, Collapse potential and Pinhole dispersivity	Atterberg limits, UCS, UU and optimum $\omega\%$ increased.	Collapse potential and maximum dry density decreased.	(Tabarsa et al., 2018)
Clay	Nanosilica	0.5, 0.7 and 1 wt%	Atterberg limits, Proctor compaction, One-dimensional consolidation, UCS and CBR	PL, compaction characteristics, UCS and CBR increased.	LL and SL decreased.	(Changizi and Haddad, 2017; Kong et al., 2018)
Loess	Nanosilica	0.2, 0.4, 0.8, 1, 1.5 and 0.2 wt%	UCS and Proctor compaction	UCS increased.	Compaction characteristics decreased.	(Kong et al., 2018)
Clay	Nanokaolinite	1, 3 and 5 wt%	Atterberg limits, Proctor compaction, UCS	Atterberg limits, maximum dry density and UCS increased.	Optimum $\omega\%$ decreased.	This study

Continued Table 1.

Type of soil	Nanoparticles/additives	Proportions	Conducted tests	Advantages		References
				Major	Minor	
Clay	Carbon nanotube (CNT)	1 wt%	Atterberg limits, Consolidation	Plasticity Index, Compressibility, Swelling Index	Hydraulic conductivity	(Taha and Ying, 2010)
Clay	Nanosilica and cement and Lime	3, 5 and 7 wt%	UCS	UCS increased.	-	(Ghazavi and Bolhasani, 2010)
Clay	Nanoclay	1, 2, 4 and 8 wt%	Atterberg limits	PI increased.	-	(Baziar et al., 2010)
Clay	Nano-Al ₂ O ₃ and Sewage Sludge ash/Cement	1, 2 and 3 wt%	Atterberg limits, UCS, swell potential and CBR	Strength properties	Swell potential and PI	(Luo et al., 2012)
Clay	Nanosilica and Lime	1, 3 and 5 wt%	UCS	UCS increased.	-	(Gelsefidi and Mamaghanian, 2013)
Silt	Nanoclay	0.5, 1, 1.5 and 2 wt%	Atterberg limits, Direct shear, UCS and CBR	Strength properties, PI, C and ϕ	-	(Nikoogar et al., 2013)
Clay	Nanoclay	1.5, 3, 4.5 and 6 wt%	UCS	UCS increased.	-	(Zahedi et al., 2014)
Silt	Nanosoil	2, 3 and 4 wt%	Atterberg limits, Direct shear and UCS	Strength properties, C and ϕ	PI	(Khalid et al., 2015)

Conclusions

The effects of adding a small amount ($<0.2\%$) of MWCNTs and CNFs to control samples indicated good improvement in Atterberg limits and a slight progress in soil compaction characteristics. Due to high surface area of MWCNTs and CNFs particles, the water adsorption is extensive, resulting in scatter variations in dry density and optimal moisture contents of the stabilized soil.

The results of the Atterberg limits experiments indicate that, by adding any percentage of nano silica, the PI reduced by around 52%. By increasing some amount of nano silica additive beyond the optimum point for soil stabilization, it causes a reduction in the overall rate of PI of the control sample. The addition of nano silica leads to an increase in water absorption and a reduction in pores between clay particles, resulting in an increase in the maximum dry density and the optimum moisture content of clay. The UCS results showed that the nano silica particles had a positive effect on hardness of the treated specimens.

The Atterberg limits obtained for the soil treated by nano kaolinite indicate that LL, PL and PI increased, intensively. It is important to note that soil treatment by 1 to 5% nano kaolinite led to enhance the dry density from 1902 to 1951 kg/m³ and reduce the optimum moisture content from 12.44 to 10.09%. The nano kaolinite particles also influenced the UCS ratios of soil by 0.73, 2.13 and 3.60, corresponding to 1, 3 and 5 wt% of additive, respectively.

The results of laboratory studies showed that the addition of 0.5 to 3 wt% nano montmorillonite significantly improved the mechanical properties of weak soil. In particular, the addition of nano montmorillonite to the control sample: increased the PI values, increased the optimum moisture content, decreased the maximum dry density and increased the UCS of treated soil.

No major development in elastic modulus (E) ratios for MWCNTs, CNFs and nano montmorillonite contents was observed. A slight increase in the E ratios was observed for nano silica particles when proportioned in 0.5, 0.7 and 1 wt% which correspond to E ratios of 1.2, 1.41 and 1.84, respectively. A better performance in E ratio is resulted from the addition of 3 and 5 wt% nano kaolinite, yielding the corresponding E ratios of 2.45 to 3.8, respectively.

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ADDITIONAL INFORMATION AND DECLARATIONS

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Competing Interests

The authors declare there are no competing interests.

Author Contributions

Abolfazl Soltani conceived the experiments, analyzed the data, prepared figures, and tables, authored or revised drafts of the paper, approved the final draft.

Amin Moradi conceived and designed the experiments, analyzed the data, contributed reagents /materials/ analysis tools, prepared figures, and tables.

Nabi-allah Ahmadi conceived and designed the experiments, analyzed the data, contributed reagents /materials/ analysis tools, prepared figures, and tables.

Data Availability

All the data are shown in the tables of this article.

Ethics Statement

The study was approved by Scientific Association of Environmental Education and Sustainable Development (EESD) <http://www.ac.ir/environment>

Supplemental Information

Supplemental information for this article can be found online at ???

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