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Original Research Article

Effect of graphene oxide nanosheets on the geotechnical properties of cemented silty soil

Farzad Naseri^a, Mohammad Irani^{b,*}, Masoud Dehkhodarajabi^c^a Department of Civil Engineering, Electronic Branch, Islamic Azad University, Tehran, Iran^b Department of Chemical Engineering, Amirkabir University of Technology (Tehran Polytechnic), Tehran, Iran^c Department of Civil Engineering, University Technology Malaysia, Johor Bahru, Malaysia

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ABSTRACT

In the present study, the effect of graphene oxide nanosheets (GO) on the geotechnical properties of cemented soil was investigated. Various concentrations of GO (0.02, 0.05 and 0.1 wt% of cement) were added to the soil to evaluate the influence of GO on the soil's compaction characteristics, consistency limits, unconfined compression strength (UCS) and direct shear parameters. The scanning electron microscopy (SEM) and X-ray powder diffraction (XRD) analysis were used to characterize the structure of synthesized GO and stabilized soil samples. The addition of GO decreased the plasticity and compressibility parameters of the treated soil samples. The tensile and the shear strength of the treated soil samples were increased with an increase in the GO concentration. The unconfined compressive strength was increased as the GO content increased in the cemented soil samples. The obtained results showed that the GO as a stabilizing agent has a considerable influence on the mechanical properties of stabilized soil.

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1. Introduction

Many soil stabilization techniques have been used to improve the geotechnical properties of the soil [1–3]. The treatment of soil was developed in the terms of strength, reduced compressibility, and hydraulic conductivity [4,5]. The cementitious additives such as cement, lime and fly ash have been widely used to enhance the geotechnical properties of various soil types [6–9]. Recently, the nano-sized materials including nanoparticles and nanotubes are most commonly used in cementitious composites to improve the mechanical

properties. The most common nano-sized materials are SiO₂, TiO₂, Al₂O₃, graphene oxide (GO) and carbon nanotubes (CNTs) [10–15]. Improvement of the properties of cement-treated soil has been mainly attributed to a soil–cement reaction, which produces primary and secondary cementitious materials in the soil–cement matrix [12].

The nanomaterials due to the high specific surface area and surface charges with fine pores, may significantly improve the physico-chemical properties of soil. Chemical stabilization of a soil by adding the nanomaterials into the soil, is one of the techniques that improve the mechanical behavior of cementitious materials-treated soil [16]. The new

* Corresponding author. Tel.: +98 021 22590926; fax: +98 021 22600600.

E-mail address: irani_mo@ut.ac.ir (M. Irani).

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stronger and stiffer matrix forms by chemical stabilization compared with original soil.

The most relevant characteristics of cementitious materials-treated soil including particles size distribution, plasticity, chemical composition and pH can be altered by chemical interactions. So, the stabilization of cementitious materials-treated soil can improve by treatment of cementitious materials with reinforcing nanomaterials [17]. The carbon nanomaterials including CNTs and GO nanosheets have been widely used to improve the cementitious material properties [18–22]. Cwirzen et al. [20] suggested adding multi-walled carbon nanotubes (MWCNTs) treatment with polyacrylic acid polymers. The highest increase in the compressive strength was obtained about 50% in cement paste by incorporation of 0.045% of the polyacrylic acid polymer-treated MWCNTs. Figueiredo et al. [16] investigated the influence of CNTs/surfactant on the mechanical properties of stabilized soil. It was verified an improvement up to 77% on the compressive strength of the material and 155% on Young's modulus by good dispersion of CNTs/surfactant into the soil sample. The obtained results indicated that a very small quantity of MWCNT improved the mechanical properties of a soil chemically stabilized with cement. However, the use of CNTs due to the poor dispersion of CNTs and weak bonding between the CNTs and the cement/soil matrix is limited.

Mohammad et al. has been investigated the transport characteristics of GO reinforced cement composites [21]. They found that GO addition to cement matrix can effectively enhance its resistance to aggressive elements by forming a strong barrier that can reduce the movement of aggressive chemicals. GO due to the high specific surface area, high intrinsic mobility and high Young's modulus leads to a remarkable enhancement in mechanical properties of cementitious material matrix [23,24]. GO sheets contain the functional groups such as hydroxyl, epoxide, carboxyl and carbonyl which facilitate the dispersion of GO in cement matrix [23,25]. Therefore, dispersion of cement is much more stable in the presence of GO nanosheets. Most research work to date has been done with GO added to cement pastes and concretes [26–28]; neglecting the study with soil matrixes. Horszczaruk et al. [27] showed that graphene oxide additive in the amount of 3 wt % in cement results in significant enhancement of Young's modulus. Pan et al. [28] studied the mechanical properties and microstructure of a graphene oxide–cement composite.

Incorporation of GO led to increase in GO–cement composite compressive strength by 15–33% and the flexural strength by 41–59%, respectively. However, the change in properties of soil by incorporation of GO into the soft soil due to the inherent characteristics of soil is impossible. So, the stabilization of cementitious materials-treated soil can improve by treatment of cementitious materials with GO.

In the present study, the GO nanosheets were added to the cement solution and following the cement/GO solution as a main agent responsible for soil stabilization was loaded into the soil skeleton matrix. Remarkably, only incorporation of 0.02, 0.05 and 0.1 wt% of GO sheets into the cemented soil samples led to increase in compressive strength by 79–127% compared with cemented soil. The mechanical behavior of the new composite materials were more studied by consistency, compaction, unconfined compression and direct shear tests.

2. Experimental

2.1. Soil samples

The soft soil samples were taken from in the Tehran province in the center of Iran. The soil sample was collected at the depth of 2.5 m and was homogenized in laboratory. The soil was composed of silt ($2 \mu\text{m} < \text{size} < 0.6 \text{ mm}$: 69%), clay ($\text{size} < 2 \mu\text{m}$: 18%) and sand ($0.6 \text{ mm} < \text{size} < 2 \text{ mm}$: 13%) particles. The properties of the studied soft soil are presented in Table 1. The unconfined compression strength (UCS) of soil was 136 kN/m^2 .

2.2. Synthesis of GO

Graphite oxide was synthesized from pure graphite powder via modified Hummers method [29]. Briefly, raw graphite and KMnO_4 were mixed in a flask containing 20 mL sulfuric acid and orthophosphoric acid under stirring at 50°C for 24 h. The prepared mixture was dispersed into the H_2O_2 (30%) and was immediately precipitated by a centrifuge at 15,000 rpm for 10 min and was washed with HCl, ethanol and deionized water several times. Finally the material was dried at 70°C for 12 h to obtain the brown GO. The chemical properties of the synthesized GO nanosheets are listed in Table 2.

Table 1 – Properties of the studied soil.

Chemical properties	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	CaO (%)	MgO (%)	K_2O (%)	pH
	66.3	16.1	6.1	1.3	0.8	2.7	3.7
Compaction properties	Maximum dry unit weight (kN/m^3)			Specific gravity	Optimum water content (%)		
	16.21			2.60	6.20		
Physical properties	Liquid limit LL (%)		Plastic limit PL (%)	Plasticity index PI (%)		Shrinkage limit (%)	
	61		32	31		19	
Mechanical properties	UCS (kPa)		Cohesion (kPa)		Internal friction angle ($^\circ$)		
	136		48		33		

Table 2 – Chemical composition of the synthesized GO.

Sample	Element percentage (%)					
	C	O	Si	Al	S	Mg
GO	60.60	35.60	1.42	0.69	1.21	0.48

2.3. Preparation of the soil/cement/graphene oxide composites

The Portland cement type I (42.5 R) of cement manufacturing company (Darab, Iran) was used in this study. The chemical properties of the cement are presented in Table 3. The amount of Portland cement used for the stabilization of the soil was 150 kilos per cubic meter of soil. The GO sheets were added in the amounts of 0.02, 0.05 and 0.1 wt% of cement. The predetermined amounts of GO nanosheets were added into the flasks containing 150 mL of water. The GO aqueous suspensions were sonicated for 20 min to obtain the homogenous solutions. The prepared GO suspensions were added into the cement solutions (the water to mixture ratio used was 4 (v/w)). Then the prepared mixture was put into the mixing bowl containing soil. The mixing was continued for 5 min to obtain the homogenous composite matrix.

In order to investigate the compression strength, the prepared mixtures were compacted into a split mold in six layers with inner diameter of 38-mm and height of 85-mm. For this, the soil samples were wrapped into the cylindrical molds for 7, 14, and 28 days at room temperature. Soil samples were prepared at maximum dry density conditions. A series of tests was conducted to evaluate the effect of the GO nanosheets on the mechanical and compressibility characteristics of soil samples.

2.4. Testing procedures

In order to estimate the maximum dry unit weight and optimum moisture of the soil samples, the compaction tests were conducted according to American Society for Testing and Materials specifications (ASTM D698) [30]. The compaction process was done in three layers. Each layer was compacted with 25 blows. The specimens were compacted in layers into a split mold of size 38-mm diameter and 89-mm height to achieve dry unit weight corresponding to maximum dry density obtained from Proctor compaction test. Each specimen was extracted from the split mold after compaction. The compaction tests were carried out for the soil samples containing 0.02, 0.05 and 0.1% of GO.

The liquid limit test of the soil samples was determined according to British Standards (BS 1377-2). The effect of GO concentration (0.02, 0.05 and 0.1% of GO by weight of cement) on consistency limits of soil samples was evaluated.

The unconfined compression tests (UCS) were carried out according to (ASTM D2166) [31] at the rate of 1.2 mm/min. Deformation values were recorded during the test. The tests were repeated at least three times and the average values were reported.

The direct shear stress test of soil samples were also evaluated according to ASTM D3080 [32]. The specimens were compacted in layers into a mold of size 6 cm × 6 cm × 2 cm to achieve a dry unit weight corresponding to the maximum dry density obtained from the Proctor compaction test. Each specimen was extracted from the mold after compaction. After curing the soil samples, normal stress (33.5, 66.8, and 121.3 kPa) was applied and soil samples were subjected at the displacement rate of 0.5 mm/min. Deformation values were recorded during the test. The cohesion and internal friction angle values of soil samples were also investigated.

The X-ray powder diffraction (XRD) pattern was recorded at 25 °C on a Philips instrument (X'pert diffractometer using Cu-K α radiation) with a scanning speed of 0.03° (2 θ) min⁻¹ to confirm the GO nanosheets structure. The morphology of GO and soil samples were characterized using a scanning electron microscopy (SEM, TESCAN, VEGA 3SB) after gold coating.

3. Results and discussion

3.1. Characterization of GO, soil/cement and soil/cement/GO

Fig. 1a shows the SEM image of the synthesized GO nanosheets with the film thickness of 20–30 nm. The size of GO nanosheets can be measured as approximately between 0.2 and 3 μ m. This indicates a randomly sizes of the GO sheets. The XRD pattern of the synthesized GO nanosheets is shown in Fig. 1b. An intense and sharp peak at 12.34° was due to the oxidization of functional groups of graphite [23].

The SEM images of soil/cement and soil/cement/GO 0.05% are illustrated in Fig. 1c and d.

The incorporation of GO sheets into the soil/cement composite can effectively force the cracks to twist around the sheet and fill some pores between the particles which resulted in particle packs with smaller pores contributing to a denser soil matrix. Similar trend is reported by Pan et al. [28] for cement containing GO.

3.2. Effect of GO nanosheets on the compactability

In order to investigate the maximum dry unit weight and optimum moisture of the soil, the cemented soil samples were treated with different contents of GO (by

Table 3 – Chemical properties of Portland cement used.

SiO ₂ (%)	Al ₂ O ₃ (%)	Fe ₂ O ₃ (%)	CaO (%)	MgO (%)	SO ₃ (%)	Cl (%)	InR (%)	L.O.I. (%)	Alkali (%)
20.40	5.25	3.95	65.5	1.2	1.05	0.03	0.62	1.40	0.60

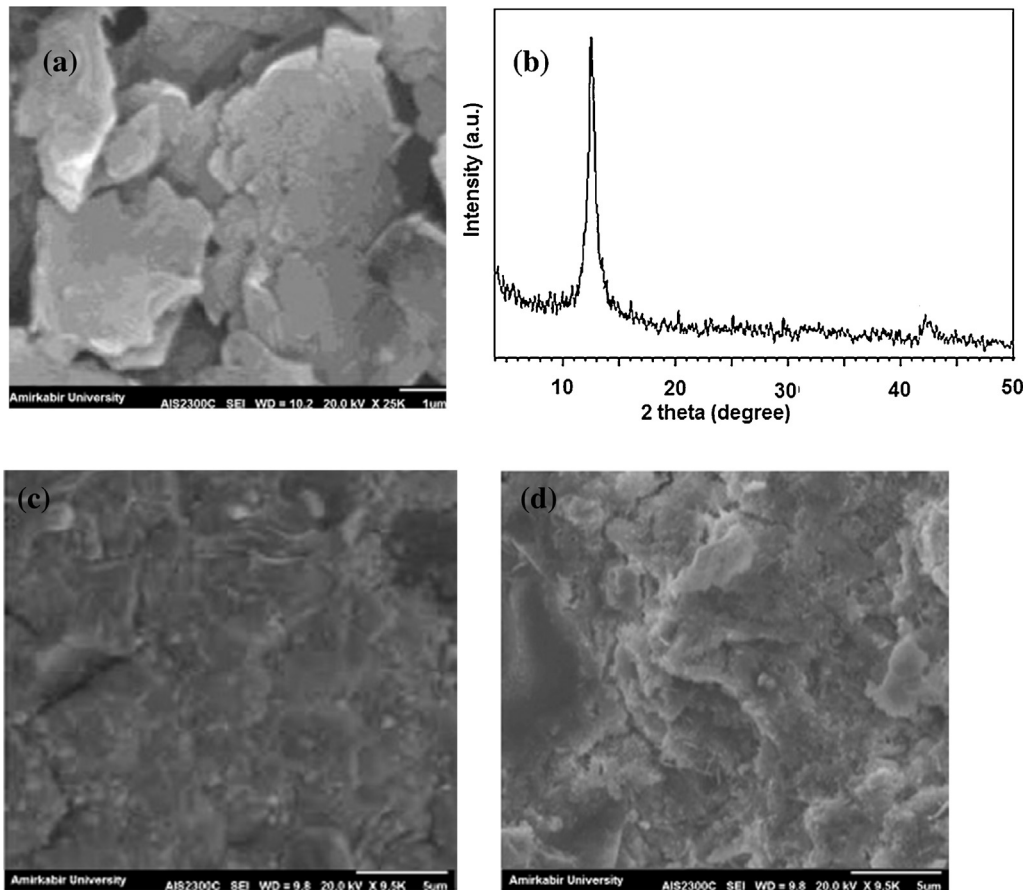


Fig. 1 – (a) SEM image and (b) XRD pattern of synthesized GO, (c) SEM image of soil/cement and (d) SEM image of soil/cement/GO 0.05%.

mass of cement). The results of compaction tests are shown in Fig. 2. It was revealed that the increasing in the GO content in the soil/cement/GO matrix resulted in enhancement of dry density and decrease in optimum moisture content. The increase in dry density can be attributed to some interactions such as C-H-S between the GO and soil samples. The existence of GO plays a role in denser formation of crystals and tendency to become denser and interwoven. Therefore, the volume of the soil sample

decreased by addition of GO in the soil/cement matrix and formed a compact structure. Similar trends are reported by other researchers [21,22]. Additionally, the interface bonding between the GO and cement and formation of C-H-S gel as well as decreasing the pores of soil samples could be responsible for decreasing the optimum moisture content.

3.3. Effect of GO nanosheets on consistency limits

The effect of GO nanosheets on the liquid limit (LL), plastic limit (PL), and plasticity index (PI) of the cemented soils was investigated which results are listed in Table 4. The plasticity

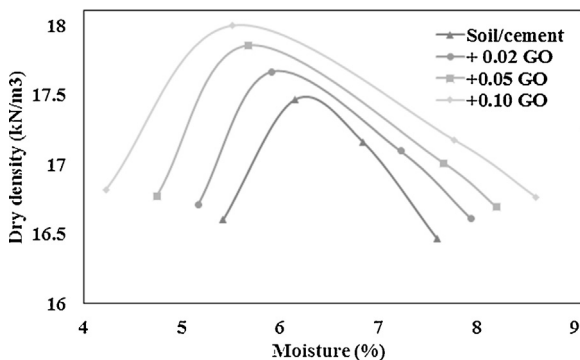


Fig. 2 – Compaction test results of soil samples.

Table 4 – Plasticity of soil samples at different GO contents.

Soil samples	Liquid limit LL (%)	Plastic limit PL (%)	Plasticity index PI (%)
Soil/cement/GO 0%	50	27	23
Soil/cement/GO 0.02%	45	24	21
Soil/cement/GO 0.05%	39	22	17
Soil/cement/GO 0.10%	36	21	15

index is defined as the difference between the LL and the PL and indicates the range of moisture contents over which the soil remains plastic. As shown, the adding of GO in the cemented soil samples changed the consistency behavior of the soil samples. Based on the obtained results, it is found that increasing of GO content resulted in decreases in LL, PL and PI values. Decrease in PL index with the presence of GO nanosheets may be attributed to the increased packing density [21]. By addition of GO in the cemented soils, the ability of the new composite material to absorb the excess water was increased which resulted in decreasing of PI by formation of the denser layers of soil samples.

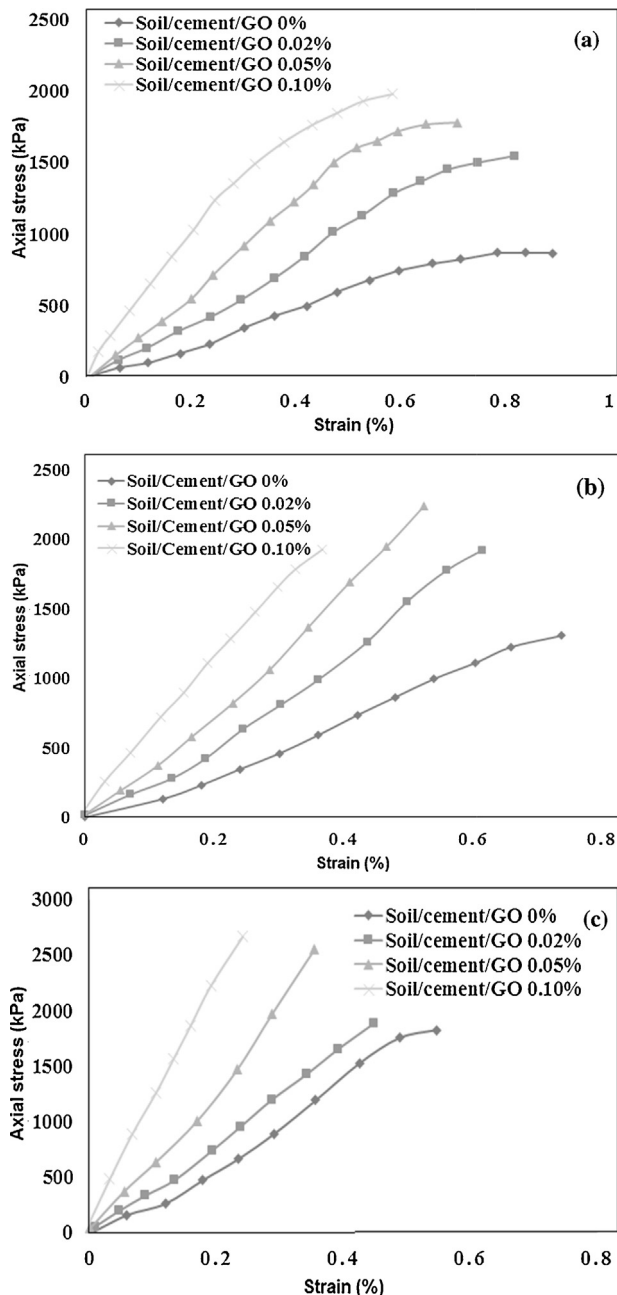


Fig. 3 – Effect of GO nanosheets on UCS of the soil samples for (a) 7, (b) 14, and (c) 28 days curing.

3.4. Effect of GO nanosheets on the compressive strength

The addition of cement to the soil increases the resistance of soil matrix and the mechanical properties of soil samples. The addition of GO to the cemented soil due to the unique properties of GO can further improve the mechanical properties of soil. The influence of GO nanosheets on the unconfined compressive strength of the soil samples for 7, 14, and 28 days curing is illustrated in Fig. 3. As shown, the incorporation of GO in the soil samples led to an increase in unconfined compressive strength of the soil samples. Additionally, increase in the GO concentration resulted in increasing of compressive strength and decreasing of strain. By addition of GO, the cemented soil samples became denser by filling some voids in the soil samples structure. Also, the good dispersion of the cement particles by addition of GO led to a better filling of free spaces between the soil particles and more resistant soil samples. By increasing the GO concentration, the soil samples became more dense and resistant in the cemented soil. Additionally, the increase in GO concentration increased the interconnection between soil particles and produced more homogenous compressible material. Therefore, the GO nanosheets had a considerable effect on increasing the unconfined compressive strength of the cemented soil samples.

3.5. Effect of GO nanosheets on the stress–strain behavior of soil samples

The shear stress–strain curves with various normal stresses (29.5, 62.5, and 120.5 kPa) of soil samples for 28 days curing are illustrated in Fig. 4. As shown, there is an increase in maximum shear strength values by addition of GO nanosheets in the cemented soil samples. Additionally, the effect of normal stress on shear strength was more prominent for the cemented soil samples with higher concentrations of GO. The sharp increase in shear strength of soil samples and following decrease in shear stress with an increase in strain were observed for all of treated soil samples under the same vertical normal stress.

By addition of GO, the matrix of cement and soil became denser, so the some voids were filled. The better filling of free spaces between soil particles and enhancement in the interconnection between clay particles with higher GO content led to produce a more homogenous material. Therefore, the treated soils exhibited more stiff behavior compared with non-treated soils. Similar trend is obtained by Azzam [33].

The cohesion (kPa) (c) and internal friction angle (ϕ) values of the soil samples after 7, 14 and 28 days curing obtained from linear regression analyses for untreated soil and treated soil samples are illustrated in Fig. 5. The correlation coefficients are almost equal to unity in the analyses ($R^2 > 0.99$). As shown, the cohesion and internal friction angle of cemented soil samples increased with increasing GO content. Furthermore, the c and ϕ values of all GO-reinforced cemented soil increased with increasing the curing time. The increase in c values could be attributed to the decreasing of voids between the soil particles and formation of compressible material. Cohesion increase more than values of internal friction angle.

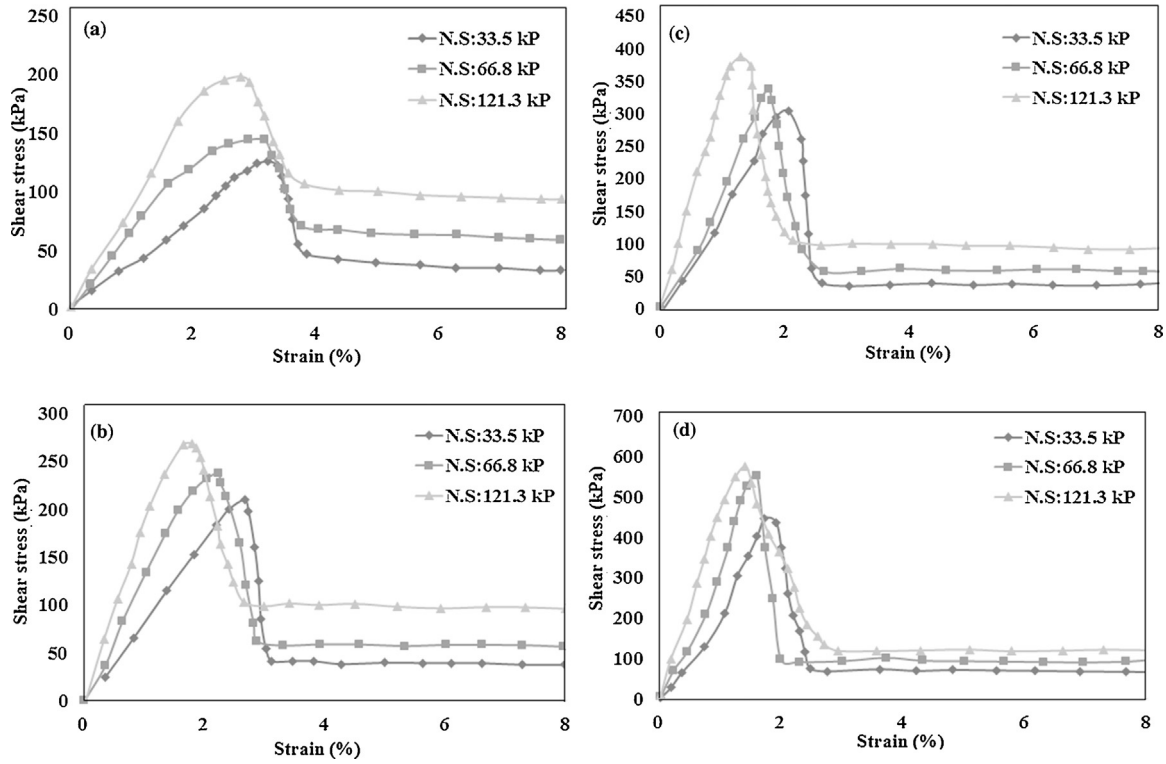


Fig. 4 – Shear stress–strain curves with various normal stresses for soil samples containing (a) 0%, (b) 0.02%, (c) 0.05% and (d) 0.10% GO.

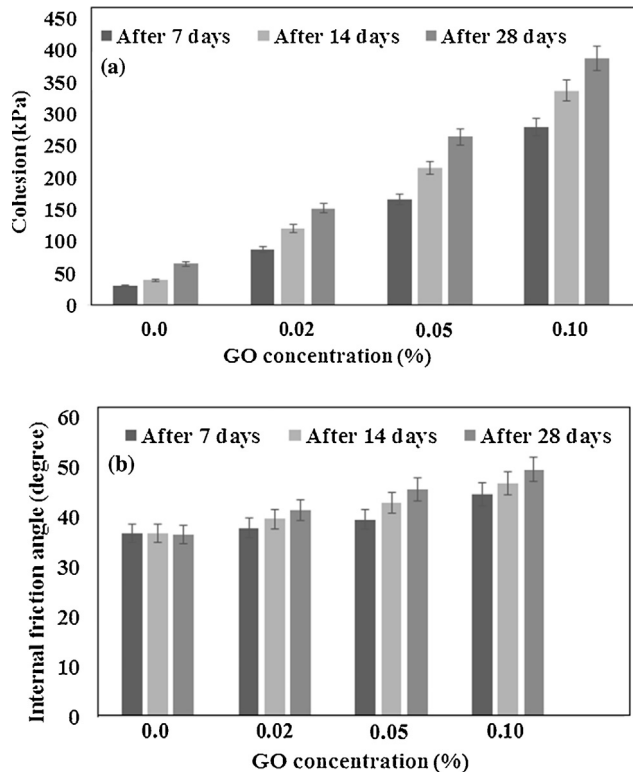


Fig. 5 – (a) Cohesion and (b) internal friction angle values of the soil samples.

4. Conclusion

The cementitious composites treated with graphene oxide nanosheets were successfully developed and were added to the Iranian soft soil samples to improve their geotechnical properties. The synthesized GO with the film thickness of 20–30 nm was produced. The SEM results of soil/cement and soil/cement/GO 0.05% indicated that the incorporation of GO into the cemented-soil resulted in decreasing the pores of soil matrix. The enhancement in GO content led to increase in dry density and decrease in optimum moisture content of treated soil samples.

The results of Atterberg limits test (including LL, PL, PI) indicated that the LL, PL and PI of the cemented soil samples were decreased with increasing GO content. The inclusion of GO nanosheets within cemented soil caused an increase in the UCS and shear strength parameters (cohesion and internal friction angle). The enhancement in the interconnection between clay particles with higher GO content led to exhibit a more stiff behavior of treated soils. The obtained results revealed that the addition of cement/GO to soil can be considered as an efficient method to improve the mechanical properties of cemented soft soil samples.

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