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Compressive and Tensile Strength of Nano-clay Stabilised Soil Subjected to Repeated Freeze–Thaw Cycles

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Abstract: Improvement of the mechanical properties of clayey soils by additional elements to enhance the strength under numerous freezing and thawing cycles has been considered as a serious concern for engineering applications in cold regions. The objective of the current study is to investigate the effect of nano-clay as a stabiliser on the mechanical properties of clay. To this end, the clay specimens were prepared by adding various percentages of nano-clay ranging from 0.5% to 3% by dry weight of soil and were experimentally tested under the uniaxial compression and tensile splitting tests under different curing times (0, 7 and 28 days) after experiencing various freeze–thaw cycles ranging from 0 to 11. It can be concluded from the results that nano-clay particles may be used as a stabiliser in geotechnical applications to improve soil property. The results indicate that the optimum moisture content (OMC) of specimens increases and the maximum dry density (MDD) decreases with the increasing nano-clay content. The specimens containing about 1% nano-clay recorded maximum values of unconfined compressive strength (UCS) as well as tensile strength. For example, the addition 1% nano-clay increased the UCS and tensile values of clay specimens under the curing time of 28 days by 34% and 247%, respectively. In addition, the long-term durability of specimens against freeze–thaw cycles increases further with the addition of nano-clay content ranging from 2% to 3%.

Keywords: Nano-clay; Compressive Strength; Tensile Strength; Freeze–Thaw Cycles.

1 Introduction

Soil stabilisation is defined as physical, chemical, mechanical, biological and combined methods which increase the stability of a soil or improve its mechanical behaviour (Asgari et al., 2015; Ornek et al., 2012; Rezaei-Hosseinabadi et al., 2021; Saadat & Bayat, 2022; Salehi et al., 2021, 2022; Sivrikaya et al., 2014; Truty & Obrzud, 2015). Nowadays, utilising inorganic salts, cationic organic polymers and nanomaterials has been significantly developed to improve the mechanical characteristics of soils, such as shear strength, durability and compressibility (Movahedan et al., 2012; Rajczakowska et al., 2015; Rajczakowska & Łydzba, 2016; Sahin & Oltulu, 2008; Taha & Taha, 2012; Turkoz et al., 2015). Nano-clays are non-toxic, low-cost materials which are produced commercially from natural clays, which come from easily accessible sources. The absence of chemical bond between layers of the clay geometry results in an increase in the capacity of stacks' layers, which leads to better absorption and dissipation of water. Nano-clays, which essentially consist of layered silicates stacked together with non-metric thickness and diameter of 50–200 nm and surfaces about 50–150 nm in one dimension and a high aspect ratio, provide interactive and well-dispersed surfaces when exfoliated, having a phyllosilicate or sheet structure. These nanoparticles are the most popular nano-reinforcements which have been usually incorporated in polymers to improve both barrier and mechanical characteristics due to their high pozzolanic activity, relatively high ion exchange capacity, high aspect ratio and economic advantages. The pozzolanic activity is a measure for the reaction rate or reaction over time between a pozzolanic material and calcium hydroxide in the presence of water. Depending on the chemical composition and nanoparticle morphology,

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nano-clays are organised into several classes such as montmorillonite, bentonite, kaolinite, hectorite and halloysite.

The features of nano-clay refer to their active interaction with other soil constituents, which is due to the high specific surface area of nanomaterials. Nano-clays are significantly refined clayey powders of mineral silicates and are used to increase the compressive and thermal strength and reduce nano-cracks of soils/mortars due to their high pozzolanic activity and also existing pollutants in the environment (Ballari et al., 2009; Changizi & Haddad, 2017; Zhang et al., 2010). Kananizadeh et al. (2011) reported that the permeability of landfills reduces by adding nano-clay. Mohammadi and Niazian (2013) explored the effect of nano-clay montmorillonite on the clay characteristics using the direct shear, unconfined compressive and California Bearing Ratio (CBR) tests. The results indicated that using the optimum amount of nano-clay (about 1.5%) results in an increase of shear strength parameters, unconfined compressive strength (UCS) and CBR. Mohammadi and Choobbasti (2018) investigated the effect of an additive with nano-montmorillonite brand on the process of self-healing on clay soil using the Atterberg limits and UCS tests. The results revealed that the addition of the additive up to 5% resulted in significant changes in the physical and mechanical characteristics of soil. The addition of nano-montmorillonite to soil increases both the plasticity index and the compressive strength. High concentration of nano-clay in older samples plays a major positive role in reduction of the dispersivity potential of soils due to high cation exchange capacity (Abbasi et al., 2018; Calabi Floody et al., 2009). Avazeh and Asakereh (2017) declared that adding nano-clay up to 2% can slightly improve the level of dispersion; however, adding excessive amounts of it increases the dispersivity potential. In addition, the applications of nano-clay in collapsible soils have been successfully approved by previous studies (Padidar et al., 2016). Using nano-clay particles as a stabiliser material in dispersive soils has attracted considerable attention due to combination of cation solutions with dispersive soils, which results in sodium ion replacement (Sameni et al., 2015). Sharo and Alawneh (2016) studied the effect of nano-clay content on the improvement of strength and swelling potential of an expansive clay using the free swelling and UCS tests. The results showed that the addition of nano-clay (about 0.1%–3.0%) may enhance the strength and decrease the swelling potential of expansive soil at low doses.

Freeze–thaw cycling, as the most frequent weathering form, changes the mechanical characteristics of geomaterials, such as strength, permeability, moisture content, stiffness and durability (Chaduvula et al., 2014; Ding et al., 2018; Kalhor et al., 2019; Konrad, 1989; Olgun, 2013; Örnek et al., 2019; Shibi & Kamei, 2014; Steiner et al., 2018). These changes occur due to the thermodynamic changes resulting from a temperature change from below 0°C to above 0°C, which results in the translocation of water molecules and ice between soil particles. As soil moisture freezes, the volume of soil increases up to about 9%, which results in a significant change in soil characteristics at the micro and macro scales (Dusenkova et al., 2013; Ghazavi & Roustaei, 2013; Konrad, 1989; Szostak-Chrzanowski & Chrzanowski, 2014). Qi et al. (2006) indicated that pure loose specimens became denser with respect to dense specimens and both loose and dense specimens had the same relative density value after they were subjected to freeze–thaw cycles. Liu et al. (2019a) carried out a series of experiments to study the influence of soil initial dry density and moisture content on the mechanical behaviour of unsaturated silty clay samples. They observed that when the degree of saturation of soil was low, pore ice was isolated during freezing. The results showed the cohesion increment after freezing results in severe frost shrinkage, which results in an increase of capillary cohesion and ice cementation. Also, when the initial degree of saturation is high, pore ice will wrap the soil particles and the generation of ice may cause cracking and expansion. Roustaei et al. (2015) carried out a series of unconsolidated, undrained, triaxial compressive tests to study the effect of polypropylene fibre on the mechanical characteristics of a clayey soil during freeze–thaw cycles. It was found that with the increase in freezing and thawing times, cohesion of clayey soil in the frozen area decreased, the internal friction angle increased and UCS reduced gradually. Zahedi et al. (2014a) mixed clay specimens with different water and nano-clay contents to investigate the influence of nano-clay on resistance of the clay soils in freezing conditions. As shown, with or without freeze-thaw cycles, adding 1.5%, 3%, 4.5% and 6% of nano-clay increased the average strength of the soil than the specimens without additive.

As seen above, previous studies have mainly investigated the effect of nano-clay on compressive strength and volume change behaviour of soils. There are limited studies on the effects of nano-clay and freeze-thaw cycles on the mechanical behaviour of cohesive soil, especially under tensile loading. In the current study, a series of unconfined compressive and tensile strength tests were performed to investigate the effect of nano-clay content, freeze–thaw cycles and curing times on the mechanical behaviour of nano-clay stabilised soil.

2 Materials and Methods

In the current study, unconfined compressive and tensile strength tests were carried out on the stabilised clayey specimens with various nano-clay contents under various freeze–thaw cycles. The soil used in this study was clayey soil, which is classified as low plasticity clay (CL) based on Unified Soil Classification System (USCS). Noticeably, the effects of freeze–thaw cycles are more considerable in fine-grain soils in comparison to coarse granular soils (Qi et al., 2006). The geotechnical properties and grading characteristic curve of the clayey soil are presented in Table 1 and Fig. 1, respectively.

The grading test showed that about 90% of soil passed in 0.075-mm sieve (number 200). Modified montmorillonite clay known as Cloisite 30B, supplied by Southern Clay Company (Gonzales, TX, USA), was used for this study. The physical properties of nano-clay are reported in Table 2. Standard proctor tests were carried out on nano-clay-soil mixtures to find the optimum moisture contents (OMCs) and the maximum dry unit weights. Fig. 2 shows the compaction curves of clayey specimens containing various nano-clay contents and the influence of nano-clay content on the OMC and the maximum dry unit weight. Fig.2b shows the normalised maximum dry density (MDD) and OMC with respect to clayey specimen without any nano-clay content. The results show that an increase of nano-clay content results in a decrease in MDD. However, OMC increases with the increase of nano-clay content. This observation is in good agreement with the previous experimental finding (Kalhor et al., 2019).

Table 1: Geotechnical properties of clayey soil.

Parameter	Liquid limit (LL) (%)	Plastic limit (PL) (%)	Plastic index (PI) (%)	G_s
Value	36	20	16	2.757

Table 2: Properties of nano-clay particles.

Parameter	Typical dry particle sizes			γ_d (g/cm^3)	G_s
	90% less than	50% less than	10% less than		
Value	10 μm	6 μm	2 μm	1.98	1.6

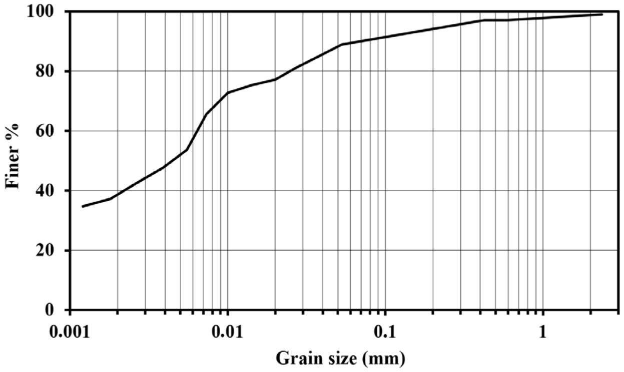

Figure 1: Grain size distribution curves of soil.

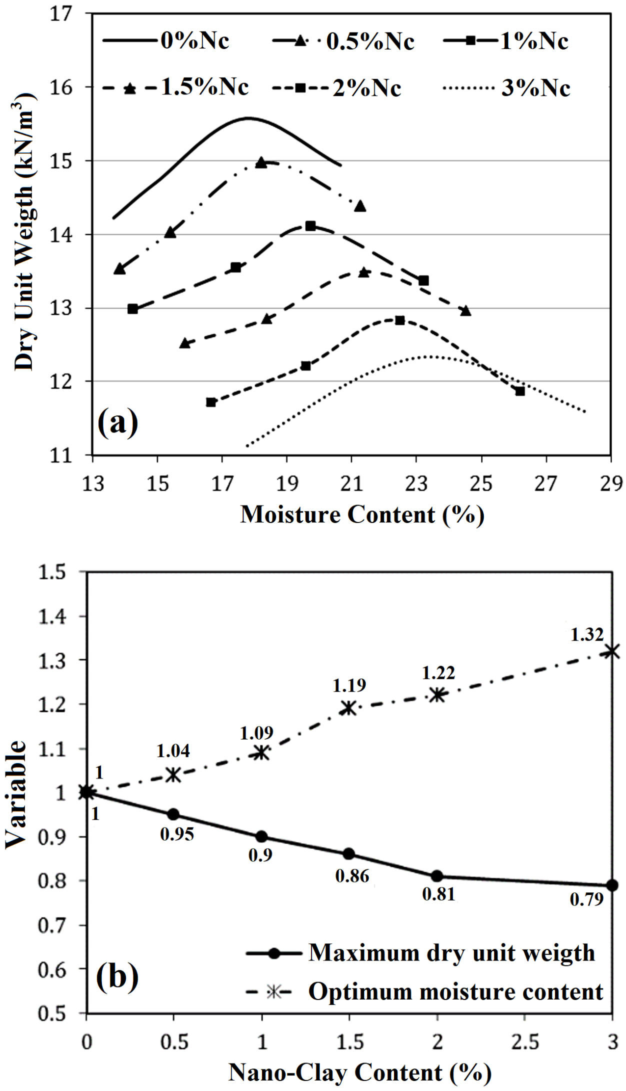

Figure 2: Influence of nano-clay on the (a) compaction curve of clay and (b) normalised maximum dry unit weight and normalised optimum moisture content.

3 Specimens' preparation and test procedure

In this study, a total of 210 split tensile strength and unconfined compression strength tests were carried out (see Table 3). It should be noted that to check the reproducibility of the results, each sample was made twice for each test condition and the results are obtained as an average of corresponding values.

The main goal of this investigation is to consider the effect of nano-clay content on both the compressive and tensile strength of stabilised specimens subjected to freeze–thaw cycles, which were prepared at the maximum dry unit weight and OMC. For this purpose, after complete drying of both soil and nano-clay particles in the oven at 110°C for 24 h, the specimens were made by adding various nano-clay contents (0%, 0.5%, 1%, 1.5%, 2% and 3% of the dry weight of the soil) to the clay soil and then OMC was added to the mixture. Substantial effort was made to ensure even dispersal of water in the mixture; clumps were broken up by hand and mixing was continued until the mixture appeared consistent throughout. For exchanging moisture among particles and forming a homogenous blend, the mixture was placed in a plastic wrap for about 24 h. The specimens were prepared by the static compaction method in a cylindrical mould of internal diameter 38 mm and height 76 mm. The exact weight of materials for each specimen was determined in accordance with the obtained maximum dry unit weight from standard compaction tests. The material was divided into four portions and each portion was compacted in a 19-mm layer. Top layers were also scratched after preparation to provide a relatively uniform joint with the upper layer and to prevent formation of weak planes. A plastic wrap was immediately used to pack the specimens after removing from the mould. After taking unit weight value into account, the specimens were placed in a curing room (maintained at 24°C ± 3°C, 95% ± 3% relative humidity [RH]). Also, for the study of effect of curing time on UCS, the specimens were tested after different curing times (i.e. 0, 7 and 28 days).

In the current study, to study freeze–thaw durability, a closed system was used. The prepared specimens were stored in a digital refrigerator at -20°C for 6 h for the freezing phase and then at +20°C for 6 h for the thawing phase, with a rate of 0.5°C per minute. This range of temperatures had been previously used in some research works (Qi et al., 2006; M. Roustaei et al., 2016). During the tests, it was observed that the height of specimens increased and decreased in freeze and thaw phases, respectively. Previous studies showed that freeze–thaw cycles up to 10 cycles have an important effect on geomaterials and after that, the effects are negligible (Ding et al., 2018; Edil and Cetin 2018; Sahlabadi et al., 2021). In the current study, all the specimens were tested under 0, 1, 3, 6, 9 or 11 cycles of freezing–thawing. It is worth noting that the specimens were exposed to freezing–thawing conditions after curing time. The curing conditions of the specimens before applying the freeze–thaw cycles were the same as the curing conditions of the specimens without exposure to freeze–thaw cycling.

The previous studies investigated the effect of nanoparticles on the mechanical behaviour of soil with nanoparticle percentages varying from 0.05% to 3% by weight (Choobbasti et al., 2019; Cui et al., 2018; Jassem & Tabarsa, 2015; Taha & Taha, 2012). To determine the effect of nano-clay contents, the specimens were prepared with five different nano-clay contents (0.5%, 1%, 1.5%, 2% and 3%). Unconfined compression tests were carried out according to ASTM-D2166 with a loading rate about 1 mm/min. The split tensile strength of specimens was determined by splitting tensile strength test using the same procedure as followed in previous studies according to ASTM C496 (Kalkan et al., 2009; Vaníček, 2013; Yilmaz et al., 2015). The tensile strength was determined as follows:

$$\sigma_T = \frac{2P_{max}}{\pi LD} \tag{1}$$

where *P* is the ultimate force during static tensile, *L* is the length of specimen and *D* is the diameter of specimen.

Table 3: Summary of the test's details.

Test group	Nano-clay content (%)	No. of freeze–thaw cycles	Curing time (days)	No. of tests
Group 1	0.5, 1, 1.5, 2 and 3	0	0, 7 and 28	15 UCS†, 15 STS‡
Group 2	0.5, 1, 1.5, 2 and 3	0, 1, 3, 6, 9 and 11	0, 7 and 28	90 UCS; 90 STS

†Unconfined compressive strength test
‡Split tensile strength

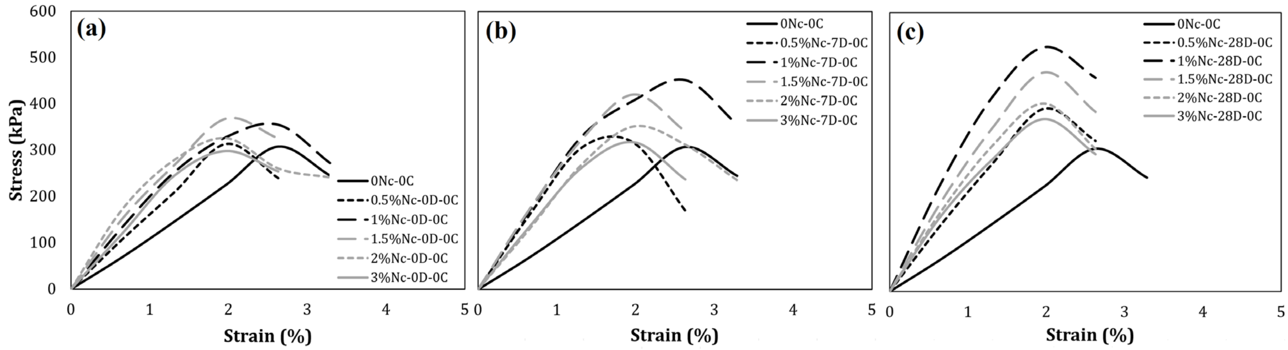

Figure 3: UCS stress–strain curves of specimens without any freeze–thaw cycles (a), curing time = 0 days (b) curing time = 7 days and (c) curing time = 28 days.

4 Results and discussion

A number of unconfined compressive and tensile strength tests were conducted on the specimens with 0%, 0.5%, 1%, 1.5%, 2% and 3% nano-clay by dry mass of the soil to evaluate the compression and tensile strength of the pure and improved clay specimens. The specimens are referred to according to the test conditions with a particular abbreviation as *n*NC-*t*D-*i*C, where *n* is the percentage of nano-clay, *t* is the curing time in terms of the day before the specimen was subjected to freeze–thaw process and *i* is the number of freeze–thaw cycles. It is worth noting that all specimens were prepared with OMC and MDD for various nano-clay contents obtained from compaction tests. The UCS tests were conducted at MDD condition. The unconfined compressive and tensile strength tests results were influenced by the combined effects of density and nano-clay content. As shown in Fig. 2, the increasing nano-clay content from 0% to 3% results in a decrease in the maximum dry unit weight and an increase in OMC.

4.1 UCS tests

Stress–strain curves of clay specimens mixed with different nano-clay content under curing times of 0, 7 and 28 days without any freeze–thaw cycles are shown in Fig. 3. As it is obvious in the results, the compressive strength increases on adding nano-clay with respect to pure clay specimen. The value of UCS increased as the nano-clay content increased up to about 1% and then decreased with increasing nano-clay content. The specimens containing nano-clay exhibited brittle fracture with a sudden drop in the stress–strain curve. In other words, the addition of nano-clay increases the stiffness and brittleness of the clay, which is due to the forming and strengthening of cementitious bonds among particles by nano-clay film. Increase in the strength of geomaterials after addition of nanomaterials like nano-clay and nano-silica has been reported in previous studies (Avazeh and Asakereh, 2017; Sharo and Alawneh, 2016; Cui et al., 2018; Hussien et al., 2023; Thomas et al., 2023).

Fig. 4 shows UCS of the specimens with various nano-clay contents under curing times of 0, 7 and 28 days. The specimens containing 1% or 1.5% nano-clay recorded the maximum values of UCS, which is due to existence of strong and sufficient bonding between clay particles and nano-clay. By adding water to clay and nano-clay mixture, through absorption of water, the nano-clay produces a viscous gel that binds the particles together, and this results in further filling of voids and increasing the strength. In fact, the interaction and adhesion force between nano-clay and clay particles lead to the formation of cementitious materials that bind the particles together. Furthermore, adding 1% or 1.5% nano-clay leads to a reduction in the distance between the clay particles, resulting in a greater number of clay particles coming into contact. In addition, increasing the curing time results in an increase of UCS, which can be attributed to the completion of long-term hardening and the formation of gels. The nano-clay content has a more pronounced effect on UCS with increasing curing time.

4.2 Tensile tests

The tensile strengths of the specimens without any freeze–thaw cycles are shown in Fig. 5. As shown in the results, the tensile strength increases with increasing nano-clay content from 0% to 1% and then decreases when the nano-clay content increases to 3%. However, the specimens containing nano-clay particles have higher tensile strength than the pure clay specimens. The presence of nano-clay in the small voids around the particles

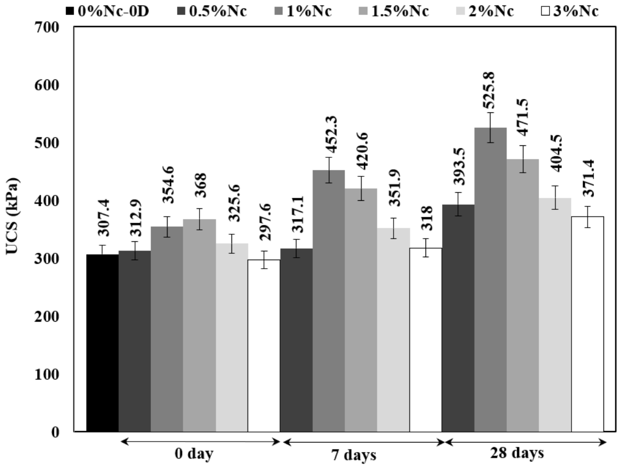

Figure 4: Unconfined compressive strength of specimens without any freeze–thaw cycles.

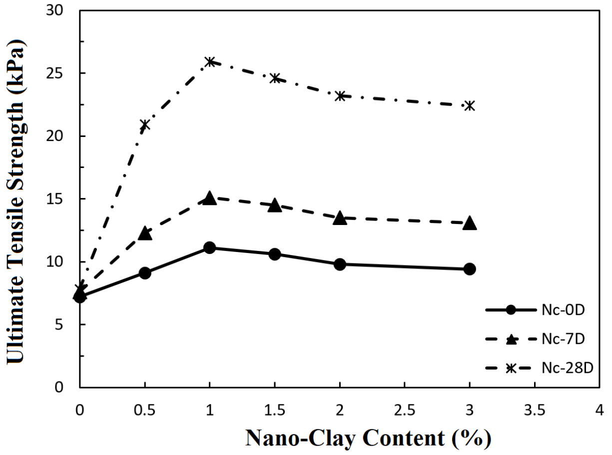

Figure 5: Tensile strength of specimens without any freeze–thaw cycles.

resulted in liquid bridges between the contact points of the particles and made the particles contact closely. For instance, the highest tensile strength was about 27 kPa, which was recorded in the specimen containing 1% nano-clay and that was cured for a period of 28 days. The tensile strength of specimen containing 3% nano-clay which was cured for a period of 28 days is about 22.5 kPa, which is higher than that of pure clay specimen (7.5 kPa). The effect of nano-clay content on the tensile strength of specimens also becomes more important with increasing curing time.

4.3 Freeze–thaw tests

The results also indicated that the effect of freezing–thawing cycles on the specimens without curing (curing time = 0 day) was more significant than on other specimens which were cured at 7 or 28 days. Fig. 6 shows the stress–strain curves of specimens without curing, which were subjected to different numbers of freeze–thaw cycles.

The normalised UCS of specimens containing nano-clay particles with respect to UCS of pure clay specimen are shown in Fig. 7. The results indicate that UCS of specimens decreases with increasing number of freeze–thaw cycles. As shown in the results, by increasing freeze–thaw cycles, the strength of specimens decreases; however, specimens containing nano-clay particles experience lower decline than pure ones. The results show that the long-term durability of specimens against freeze–thaw cycles increases further with the addition of nano-clay content ranging from 2% to 3%. The reduction of strength of stabilised specimens due to freeze–thaw cycles has also been observed in previous studies (Bozbey et al., 2018; Jafari & Esna-ashari, 2012; H. Liu et al., 2020; Pan et al., 2023; Waleed et al., 2023).

Fig. 8 shows the normalised tensile strength of specimens which were subjected to different cycles of freeze–thaw. Increasing freeze–thaw cycles results in a decrease in the tensile strength of specimens. It was observed that the results of tensile strength tests are same as the results of UCS tests, so the strength reduction of stabilised specimens is lower than that of pure ones. The lowest reduction in ultimate tensile strength due to freeze–thaw cycles was observed in the specimens with a nano-clay content of 2% or 3%. The specimens containing 2% or 3% of nano-clay had the least reduction in strength at the time of applying the freeze–thaw cycles. In other words, during the freezing process, the pore volume in soil increases due to the phase transformation of pore water into ice. The process changes the void ratio and alters the structural characteristics at the macro and micro scales. The reduction in compressive and tensile strength in nano-clay stabilised specimens depends on increasing distance between the soil particles due to formation of ice lenses below 0ºC after the freeze–thaw action. The ice force causes the clay particles to separate from each other. As shown in Figs 7 and 8, the reduction in compressive and tensile strength decreases with increasing curing time for the stabilised specimens. On the other hand, the initial cycles of freezing–thawing have a significant effect on the reduction in compressive and tensile strength. This shows that the temperature shock in the first cycle results in an important distribution of the soil fabric.

The results show that the decrease in compressive and tensile strength in samples without curing (curing time = 0 day) is lower than in other specimens (cured at 7 and 28 days). The freeze–thaw cycles resulted in destruction

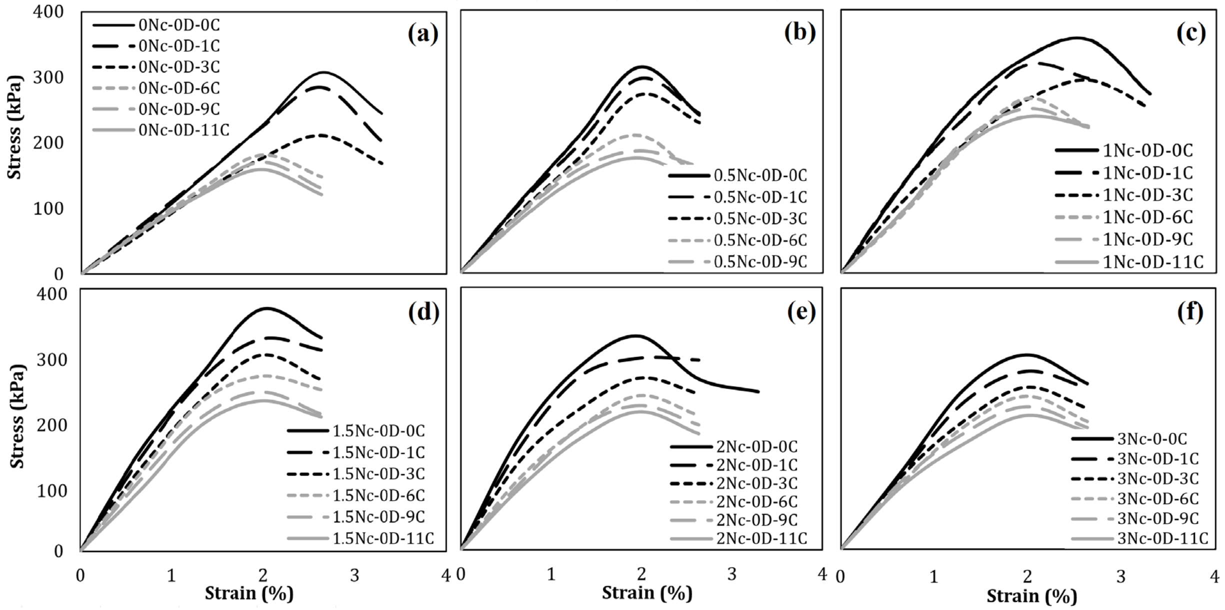

Figure 6: UCS stress–strain curves of specimens subjected to freeze–thaw cycles: (a) nano-clay content = 0%, (b) nano-clay content = 0.5%, (c) nano-clay content = 1%, (d) nano-clay content = 1.5%, (e) nano-clay content = 2%, (f) nano-clay content = 3%.

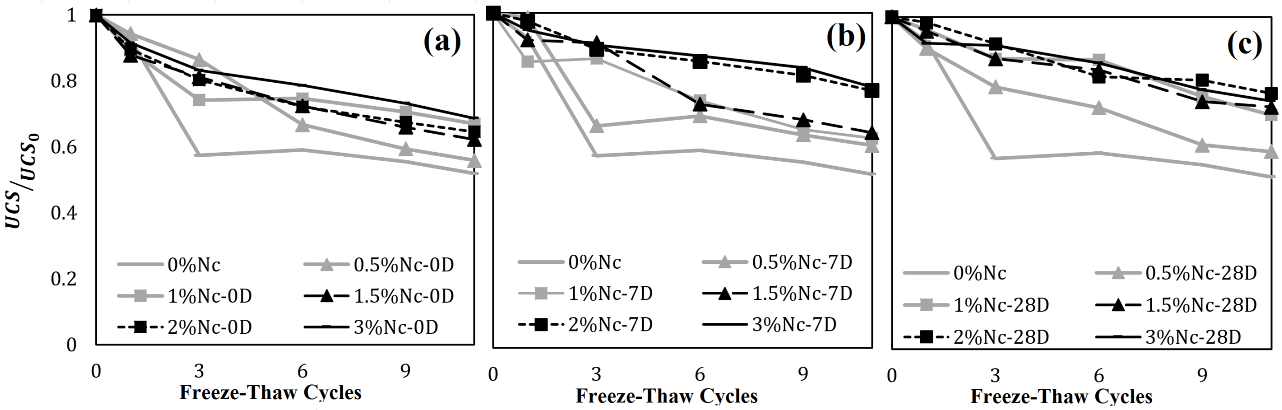

Figure 7: Normalised unconfined compressive strength of the specimens versus number of freeze–thaw cycles: (a) curing time = 0 day, (b) curing time = 7 days, (c) curing time = 28 days.

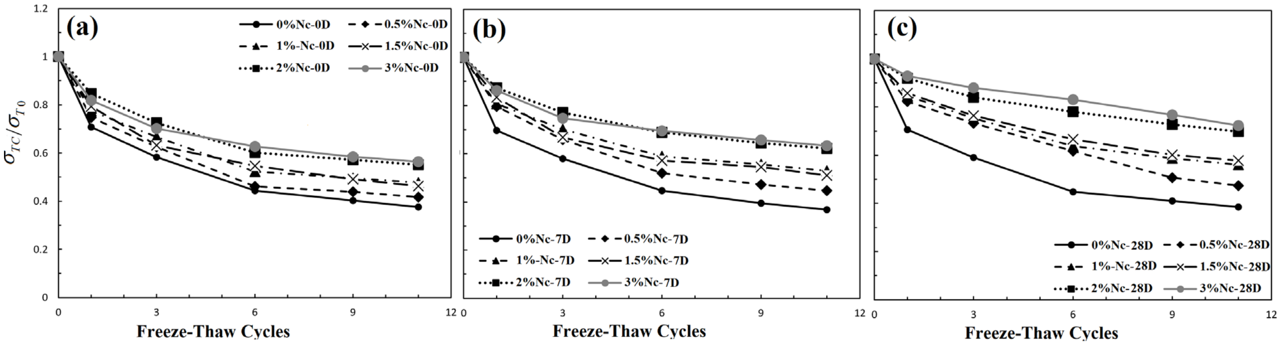

Figure 8: Normalised ultimate tensile strength of specimens versus number of freeze–thaw cycles: (a) curing time = 0 day, (b) curing time = 7 days, (c) curing time = 28 days.

of the cement bonds and ultimately caused a decrease in the strength.

5 Conclusions

In this study, improvement of strength characteristics of clayey specimens with nano-clay particles was studied under different additive contents, curing times and number of freeze–thaw cycles. The results of this research can be used in the improvement of cohesive soils in construction projects. After testing about 210 specimens through unconfined compressive and tensile strength, the following results can be drawn:

1. Based on the stress–strain curves of the specimens without any freeze–thaw cycle, it can be conducted that adding nano-clay to soil specimens results in an increase of stiffness; however, the maximum values can be achieved in nano-clay content about 1%. In early age specimens, it was observed that the highest UCS was obtained at 1.5% nano-clay content and further addition of nano-clay reduced the strength, whereas by increasing the curing time, the highest UCS was obtained in the specimens containing 1% nano-clay.
2. The optimum nano-clay content for increasing compressive and tensile strength is about 1%. After 28 days of curing, the compressive and tensile strengths of the specimen containing 1% are more than 1.7 and 3.4 times of pure clay specimen, respectively. Increasing the curing time results in a significant increase in both compressive and tensile strengths of the stabilised specimens.
3. For the specimens subjected to freeze–thaw cycle, the unconfined compressive and tensile strength values of pure clay specimens decreased significantly by increasing the number of freeze–thaw cycles. Also, a dramatic drop in the strain corresponding to the maximum compressive stress was observed for the specimens subjected to the six freeze–thaw cycles.
4. Specimens containing nano-clay content of 0.5% and 1% showed a similar trend to the pure clay specimen; however, all the maximum strengths occurred at a given strain and the amount of strength degradation decreased by increasing the nano-clay content. The specimens containing about 2%–3% nano-clay showed less unconfined compressive and tensile strength degradation after they were subjected to the freeze–thaw cycles. In other words, the specimens containing about 2% to 3% nano-clay had better response against the freeze–thaw cycles than other specimens.
5. The effect of freeze–thaw cycles on reducing the strength of the specimens decreases with increasing curing time. The effect of freeze–thaw cycles on the strength degradation of the stabilised specimens decreases with increasing curing time.

Data availability statement Data availability statement: All data that support the findings of this study are included within the article.

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