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Mechanical Performance and Freeze–Thaw Durability of Expansive Clay Stabilized with Graphene Oxide and Fly Ash: A Laboratory Study

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ABSTRACT

Expansive clay soils are characterized by their high-water affinity and significant volume changes, which frequently result in structural issues such as swelling, settlement, and cracking, particularly under freeze–thaw (F–T) conditions. This study investigates a dual-stabilization method using fly ash (FA: 5–15%) and graphene oxide (GO: 0.05–0.15%) to enhance the mechanical strength and durability of such soils. After 28 days of curing, samples underwent 3, 6, and 9 F–T cycles, followed by unconfined compressive strength (UCS) testing. Results show that the GO–FA combination significantly improved soil performance, with the optimal mix (10% FA + 0.1% GO) achieving a 76% increase in UCS at zero cycles and reducing strength loss after nine cycles by over 45% compared to untreated soil. These outcomes demonstrate the promise of GO–FA stabilization as a sustainable and effective solution for expansive soils in cold-region geotechnical engineering.

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1. Introduction

Freeze-thaw cycles are among the most important environmental phenomena in cold and temperate regions, directly and significantly affecting the geotechnical behavior of various types of soils, particularly fine-grained soils with high swelling potential. These cycles occur when the ambient temperature alternately passes the freezing point of water, leading to the freezing and then successive thawing of water within the soil mass. As a result of this process, significant changes are created in the microscopic structure, physical and mechanical properties, as well as the engineering performance of the soil.

Expansive soils, such as clay soils with a mineral structure of montmorillonite or illite, have a high capacity for water absorption and volume change due to their specific mineralogical nature and are prone to expansion and contraction behaviors. Under the influence of freeze-thaw cycles, the water in the pores of these soils freezes and increases in volume by about 9%, creating internal stresses in the soil. These stresses can lead to structural failure within the soil, the development of microcracks, and, in the long term, to a reduction in soil integrity. Furthermore, during the thawing phase, the entry of water into the porous soil system, together with a relative decrease in dry density and weakening of interparticle

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bonds, reduces the shear and compressive strengths of the soil [1-4]. Experimental evidence from cyclic tests under controlled conditions has shown that after only a few freeze-thaw cycles, shear strength parameters such as the angle of internal friction and cohesion decrease significantly, and the potential for sudden settlements or inelastic deformations in the soil increases.

From a geotechnical engineering perspective, the consequences of this process on the performance of structures built on such soils are significant. For example, in infrastructure such as pavements, railways, surface foundations, and buried piping systems, volume changes due to freezing can lead to local swelling (heave) and then thaw settlement, which over time can cause cracking, misalignment, or costly failures. These effects are more severe in expansive soils because water absorption before freezing is greater, and a higher volume change capacity is created in the cycles. Also, structural erosion caused by the aforementioned cycles reduces the initial impermeability, increases the effective porosity, and ultimately facilitates the movement of water and chemically aggressive substances within the soil. Considering the different dimensions of the impact of these cycles, improvement and modification of the behavior of expansive soils exposed to freezing is an undeniable necessity in civil and infrastructure projects [5-9]. One common method is to use chemical additives such as lime, cement, fly ash, or nanomaterials that, by creating pozzolanic or cementation reactions, strengthen the bonds between soil particles and reduce their sensitivity to temperature and humidity changes [10-12]. Fly ash is a by-product of coal combustion in thermal power plants and has pozzolanic behavior due to its high content of silica (SiO_2), alumina (Al_2O_3), and calcium oxide (CaO). Pozzolanic reactions between fly ash and soil compounds, especially in the presence of water, lead to the formation of cementitious compounds such as hydrated calcium silicate (C–S–H) and hydrated calcium aluminate (C–A–H), which increase the adhesion of soil particles, reduce permeability, and improve the compressive and shear strength of the soil. In addition to mechanical properties, fly ash changes the soil structure from a dispersed state to a compact and cementitious structure by reducing the cation exchange potential and surface water absorption by clay particles. Additionally, due to the alkaline nature of fly ash, the soil pH increases, providing a suitable environment for accelerating pozzolanic chemical reactions. From an environmental and economic perspective, the use of fly ash as a substitute for expensive chemicals such as lime or cement has significant advantages. As an industrial waste, this material causes environmental pollution if not used properly. However, if scientifically exploited in soil improvement projects, not only are geotechnical problems solved, but also a sustainable use cycle of waste materials is established [13-17].

In recent decades, the emergence of nanotechnology, and especially the application of carbon nanomaterials such as carbon nanotubes (CNTs), graphene and its derivatives, and carbon black nanoparticles, has opened new horizons in advanced soil improvement. Due to their unique physical and chemical properties, including very high specific surface area, high Young's modulus, significant thermal and electrical conductivity, and remarkable chemical stability, carbon nanomaterials have the ability to interact effectively with soil particles. When these nanomaterials are added to soil, based on microscopic and spectroscopic studies, it has been observed that they increase structural cohesion, reduce permeability, and improve soil shear strength by creating physical and chemical bonds with clay particles. One of the most important mechanisms of action of carbon nanomaterials is to fill the empty space between soil particles and strengthen interparticle bonds [18-22]. Carbon nanotubes penetrate the space between fine soil particles and act like reinforcing strips, reducing deformability and increasing soil hardness. On the other hand, the presence of carbon nanomaterials can accelerate the hydration process of cement or other additives, and when combined with pozzolanic materials, improve the chemical reactions of geopolymers [23-25]. Graphene Oxide (GO) is one of the newest carbon nanomaterials that has been considered as an innovative additive in engineering soil improvement in recent years. This material, which is an oxidized derivative of graphene and has a two-dimensional structure with active functional groups such as hydroxyl, epoxy, and carboxyl, has the ability to interact effectively with soil particles due to its high specific surface area, suitable polarity, and stability in aqueous environments, and can cause significant changes in their physical and mechanical properties [26-29].

Numerous studies have shown that adding very small amounts of graphene oxide leads to a significant improvement in compressive and shear strength, reduced permeability, improved compressibility, and increased volumetric stability in expansive clay soils. The mechanism of action of graphene oxide is based on increasing interparticle bonds and creating reinforcing microscopic networks between soil particles. This nanomaterial, by being placed in the space between fine-grained particles, increases adhesion and cohesion between them and transforms the dispersed structure of clay soils into a flocculated and dense structure. The presence of active functional groups on the surface of graphene oxide provides suitable reactivity conditions for the formation of hydrogen and ionic bonds with the surface of soil particles, which results in increased hardness, elastic modulus, and chemical stability of the soil. In addition, due to the polar

Table 1.
Summary of research on the role of fly ash and graphene oxide in soil improvement

R	Year	Type of additive	Percent of additive (%)	Results	References
1	2004	Fly ash	0 – 46	Fly ash addition lowers soil dry density, increases void ratios and porosity, and enhances shear strength nonlinearly	[35]
2	2005	Fly ash	0 – 20	High calcium fly ash forms tobermorite, enhancing the density and stability of the soil	[36]
3	2006	Fly ash	0 – 20	Due to the lower specific gravity of the fly ash than that of the soil, the maximum dry density decreased, and the optimum moisture content increased with increasing fly-ash content	[37]
4	2007	Fly ash	0 – 16	The compressive strength of sludge ash/soil was less than fly ash/soil. The bearing capacities for both fly ash/soil and sludge ash/soil were five to six times and four times, respectively, higher than the original capacity	[38]
5	2012	Fly ash	0 – 25	The UCS and CBR values increased by adding optimum fly ash content and lime	[39]
6	2015	Graphene oxide (GO)	0 – 0.5	GO altered the morphology of geopolymers from a porous nature to a substantially pore-filled morphology with increased mechanical properties	[40]
7	2016	Fly ash	0 – 10	Bearing capacity of soft soil can be improved substantially, and swell can be reduced significantly by using Class C fly ash	[41]
8	2016	Graphene oxide (GO)	0 – 0.1	GO reduced soil plasticity and compressibility while increasing tensile and shear strength of the treated soil	[42]
9	2017	Graphene oxide (GO)	0 – 0.1	The addition of GO into the soil generally decreased the soil's void ratio under a given hydrostatic consolidation pressure, while increasing its undrained shear strength	[43]
10	2018	Fly ash	0 – 50	Incorporation of biocement in fly ash It is an effective means of increasing the strength of expansive soils	[44]
11	2020	Fly ash	0 – 9	When Microbially induced carbonate precipitation (MICP)-treated sand mixed with Fly ash, the deviator stress increased, caused by the bonding of precipitated $CaCO_3$ in MICP	[45]
12	2021	Graphene oxide (GO)	0 – 0.12	The SEM results showed that due to the synergistic filling effect and hydration effect of cement/GO, the microstructure was denser, and the characteristics of cracks were refined	[46]
13	2021	Graphene oxide (GO)	0 – 0.1	As the curing time of graphene oxide increases, the soil compressibility (Cc and Cs) decreases. The coefficient of consolidation (Cv) decreases as the curing time of graphene oxide increases	[47]
14	2023	Graphene oxide (GO)	0 – 0.1	The addition of graphene oxide significantly improved the UCS with a substantial reduction in failure strains, showing relatively brittle behavior	[48]
15	2023	Fly ash	0 – 30	Increasing the sawdust ash/high calcium fly ash (HCFA/SDA) mixture and cement content enhanced expansive soil properties while lowering the liquid limit (LL) and plasticity index (PI)	[49]
16	2024	Graphene oxide (GO)	0 – 0.1	SEM test results reveal that due to nucleation effects, GO promotes the generation of hydration product C-S-H, enhancing the resistance of cement soil samples to external salt erosion	[50]
17	2024	Graphene oxide (GO)	0 – 0.05	GO improves stiffness and reduces energy consumption and damping ratio in modified coastal cement soil	[51]
18	2024	Graphene oxide (GO)	0 – 0.06	The effect of GO and its strong bonding with the cement matrix significantly improved the microstructure of the specimens by reducing pores and defects	[52]
19	2024	Graphene oxide (GO)	0 – 0.1	The GO-incorporated hydrophobic geopolymer exhibits a compressive strength comparable to the unmodified geopolymer	[53]
20	2025	Graphene oxide (GO)	0 – 0.02	GO improved the compressive strength of alkali-activated fly ash-based geopolymer and reduced the decrease in compressive strength over freeze-thaw cycles	[54]

and highly hydrophilic properties of graphene oxide, this material absorbs and stabilizes water in the micropores of the soil and significantly prevents the swelling and shrinkage of soils sensitive to moisture changes [30-34].

Given the positive properties of fly ash and graphene oxide in soil improvement, researchers have drawn attention to studying and evaluating their role in improving the geotechnical properties of problematic soils, especially fine-grained soils. Table 1 summarizes the most important

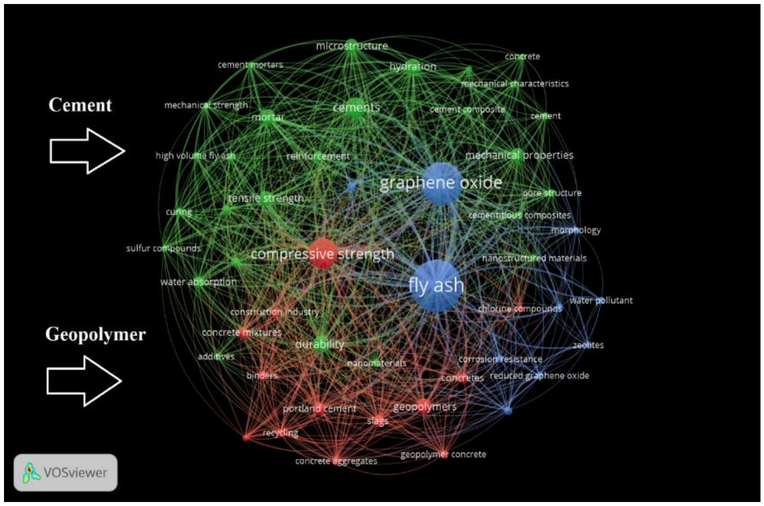

Fig. 1. Co-occurrence analysis of scientific terms in studies of GO and fly ash related to the construction field

research conducted on the effect of fly ash and graphene oxide on the geotechnical properties of soils. This table includes information on the type of additive and the optimal percentage of the material used, and the key results of each study.

In recent years, with the exponential growth of scientific production in various fields, the need for analytical tools to better understand the structure, process, and impact of scientific studies has increased. Bibliometric analysis, as one of the most important quantitative methods in the evaluation of scientific and research information, allows for the systematic examination of scientific publications, collaboration patterns, citation networks, and conceptual evolution in a specific field. This type of analysis, using bibliographic data extracted from reliable databases such as Web of Science, Scopus, or Dimensions, allows researchers to identify and analyze the dynamics of knowledge production, key researchers, leading institutions, and emerging topics. One of the prominent and widely used tools in the field of bibliometric data analysis and visualization is the VOSviewer software, developed by the Center for Science and Technology Studies at Leiden University in the Netherlands. This free software, using clustering and network drawing algorithms, has a high ability to graphically display relationships between data. VOSviewer's main capabilities include drawing co-authorship maps, co-occurrence maps, co-citation maps, and bibliographic coupling maps, each of which displays some type of relationship between bibliographic elements such as authors, institutions, countries, keywords, and articles.

Fig. 1 is the output of bibliometric analysis using VOSviewer software, which displays a network of synonyms in the field of research related to fly ash and graphene oxide, based on data extracted from scientific databases. In this map, each node represents a frequently occurring keyword in scientific texts, and the size of the nodes is determined in proportion to the frequency of use of that word in different articles. The links between nodes represent the intensity of their co-occurrence in a document, and the color clustering is the result of the VOSviewer clustering algorithm, which places related topics together based on conceptual relationships. In this network structure, three main color clusters can be seen: the blue cluster, which is centered around keywords such as fly ash, graphene oxide, cementitious composites, nanostructured materials, and reduced graphene oxide, and represents the field of research related to nanomaterials and new cementitious compounds; The green cluster, which includes concepts such as microstructure, hydration, cements, mechanical strength, tensile strength, and cement mortars, indicates the focus of research on microscopic behavior, hydration processes, and mechanical properties of mortars and cement; and the red cluster, which includes concepts such as compressive strength, durability, geopolymers, concrete aggregates, slags, and recycling, focuses on the field of durability, the use of alternative materials, recycled concrete, and geopolymer systems. A careful examination of the position of graphene oxide and fly ash in this map shows that these two terms not only have the highest frequency and the highest connection with other concepts, but also play a role as the central core of many researches in the field of modern building materials. The strong connection of these two terms with terms such as compressive strength, cementitious composites, durability, nanomaterials, and mechanical properties indicates the extensive focus of scientific studies on their application in improving the physical and mechanical properties of building materials. However, it is very striking in this analysis that throughout this conceptual network, no terms related to soil, geotechnical engineering, soil stabilization, clay soils, or concepts related to soil improvement are seen.

This apparent absence in the map vocabulary indicates that despite the widespread use of these two materials in the field of concrete, cement mortars, and even geopolymer compounds, their application in the field of soil improvement has not been seriously and organizedly reflected in the scientific literature. On the other hand, concepts such as geopolymer and geopolymer concrete, which are in the red cluster, although they are present in the map, the relatively small size of the nodes and their peripheral position in the network structure indicate that this field is still in the early stages of growth and development and has not yet reached the stage of study saturation. This indicates that research on geopolymers, despite their high potential, is still in a secondary position compared to studies on traditional cementitious systems. Overall, the presented analysis of this bibliometric picture reveals that although the application of fly ash and graphene oxide in the cement and concrete industry has reached relative maturity and has occupied a large part of the scientific literature, areas such as geopolymers and especially geotechnical engineering are still not saturated in terms of research, and this issue can be considered as a serious scientific gap and at the same time a valuable research opportunity to expand the frontiers of knowledge in the future.

2. Significance and Novelty of the Study

Expansive soils pose one of the most persistent challenges in geotechnical engineering due to their severe shrink–swell behavior under seasonal moisture fluctuations. This behavior often results in structural instability, foundation damage, and costly maintenance in infrastructure projects, particularly in cold regions where freeze–thaw cycles exacerbate the problem. While various chemical stabilizers have been used to improve the strength and deformation characteristics of expansive clays, their long-term performance under harsh environmental conditions remains a critical concern. Therefore, exploring durable, cost-effective, and environmentally responsible additives has become a key research priority in sustainable ground improvement.

This study introduces a dual-stabilization approach using graphene oxide and fly ash, aiming not only to improve the mechanical behavior of expansive clay but also to enhance its freeze–thaw durability, an aspect that has received limited attention in the literature. The novelty of this work lies in the systematic evaluation of UCS, elastic modulus (E50), and stress–strain responses under successive F–T cycles, which provides a comprehensive understanding of both strength enhancement and long-term degradation behavior. Furthermore, the use of nano-scale GO in combination with industrial by-product FA offers a promising synergy between high-performance nanomaterials and sustainable geotechnical practices.

3. Materials used

3.1. Soil

In this research, a type of expansive clay soil was selected, which is widely recognized for presenting critical challenges in geotechnical applications. Its tendency to undergo considerable volumetric changes upon moisture variation makes it highly susceptible to shrinkage and swelling, potentially compromising the stability of overlying structures. The particle size distribution of the selected clay was analyzed based on ASTM D422 standards [55], and its key physical properties are summarized in Table 2.

Table 2.
Physical characteristics of the expansive clay studied

Property	Value	Unit	Standard
Gs	2.65	Dimensionless	ASTM D854 [56]
LL	146.2	%	ASTM D4318 [57]
PL	43.9	%	ASTM D4318 [57]
PI	102.3	%	ASTM D4318 [57]
MDUW	14.7	KN/m³	ASTM D698 [58]
OMC	24.6	%	ASTM D698 [58]

3.2. Graphene oxide powder

Graphene oxide has been utilized as a nanomaterial to enhance the engineering properties of expansive soil in the present research. Due to its unique two-dimensional structure, high specific surface area, and the presence of active functional groups, graphene oxide has the potential to improve the soil's mechanical properties and reduce its swellability. This material interacts with soil particles and modifies the soil structure through physical and chemical bonding. The specifications of the graphene oxide used in this study are presented in Table 3.

Table 3.
Characteristics of graphene oxide used

Specification	**Unit**	**Value**
Appearance	-	Powder
Color	-	Grey - Black
Density	(g/cm3)	1.9-2.2
Specific Surface Area	(m^2/gr)	1000-15000
Abbreviation	-	GO

3.3. Fly Ash powder

In this study, a processed fly ash known commercially as Pozzocrete 63, manufactured by Dirk India Pvt. Ltd., was used. This artificial pozzolanic material is derived from coal-fired power plants and undergoes industrial treatment to produce a finely divided, uniform powder. Its chemical composition is rich in silica and alumina, contributing to its favorable pozzolanic reactivity. These properties enhance the bonding within the cementitious matrix, reduce permeability, and improve the mechanical performance of the final product. Due to its consistent physical characteristics and controlled quality, Pozzocrete 63 is considered a suitable additive in various civil engineering applications, including soil stabilization, special concretes, and engineered mortars.

3.4. Sample Preparation and Experimental Program Design

The primary objective of this study is to evaluate the strength improvement of expansive clay through stabilization using fly ash (FA) and graphene oxide (GO). For specimen preparation, the soil was first oven-dried and sieved through a No. 40 mesh. Then, fly ash was blended with the dry soil at predetermined weight percentages. Based on the results of the Standard Proctor compaction test, the optimum moisture content was gradually added to the mixture in stages, followed by mechanical mixing for 10 minutes. This process was repeated for samples containing varying percentages of graphene oxide as well as for combined mixtures of FA and GO. The blended materials were then used to fabricate cylindrical specimens with a diameter of 38 mm and a height of 76 mm. Each specimen was compacted in four layers inside lubricated steel molds, with each layer receiving four uniform blows. After compaction, the specimens were carefully demolded and immediately sealed in plastic bags to preserve moisture and allow for 28 days of curing under controlled conditions. Unconfined compressive strength (UCS) tests were performed in accordance with ASTM D2166 [59]. To evaluate the influence of freeze–thaw cycling, the cured samples were subjected to 3, 6, and 9 cycles using a specialized freezer.

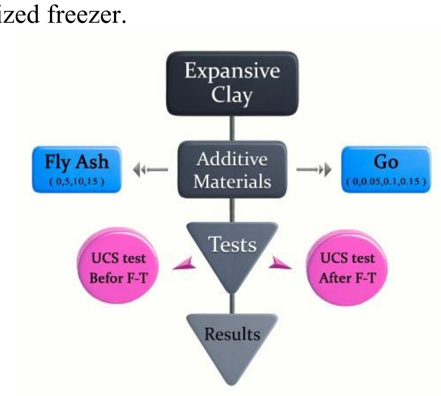

Fig. 2. Schematic representation of the experimental procedure followed in this study

For the freezing stage, the specimens were placed at temperatures ranging from −15°C to −20°C, while thawing was conducted at 20°C or ambient temperature [60,61]. The mix designs and experimental variables are summarized in the following tables. The tested FA contents were 5%, 10%, and 15% by weight, and GO contents were 0.05%, 0.10%, and 0.15% by weight. These percentages were selected based on typical ranges reported in the literature. The overall experimental procedure is illustrated in Fig. 2.

4. Results and Discussion

4.1. Improvement of UCS Using GO and FA under F–T Cycles

The variations in unconfined compressive strength (UCS) of the samples containing graphene oxide (GO) and fly ash (FA) under different freeze–thaw cycles are presented in Fig.3. According to the results, at each fixed percentage of fly ash, increasing the amount of graphene oxide leads to a noticeable improvement in compressive strength. This trend suggests that GO effectively contributes to the mechanical reinforcement of the soil by enhancing interparticle bonding and improving structural cohesion. Among all the tested combinations, the sample with 10% fly ash and 0.1% graphene oxide exhibited the highest strength, showing an increase of approximately 76% compared to the untreated expansive clay. This superior performance can be attributed to the synergistic effect of the pozzolanic activity of fly ash and the stabilizing action of graphene oxide, which together enhance interparticle adhesion and reduce detrimental porosity. As the number of freeze–thaw cycles increases, a gradual reduction in strength is observed across all specimens, which is a typical behavior in soils subjected to thermal cycling. Repeated freezing and thawing induce volumetric stresses within the soil mass, leading to the degradation of mechanical bonds and partial disintegration of the compacted structure. However, the inclusion of GO and FA mitigates the severity of this decline. The slower rate of strength loss in treated samples suggests that the additives play an important role in limiting the damage induced by freeze–thaw actions [53,62].

4.2. Stress–Strain Behavior of Optimized Soil Under Freeze–Thaw Cycles

Fig. 4 shows the stress–strain curves of the optimized soil samples subjected to different freeze-thaw cycles (0, 3, 6, and 9 cycles). These curves are used to examine the failure mode and ductility of the samples. At cycle zero, the curves exhibit more brittle behavior characterized by high peak strength and low failure strain. With an increasing number of cycles, the curve characteristics gradually change, and the material behavior tends to

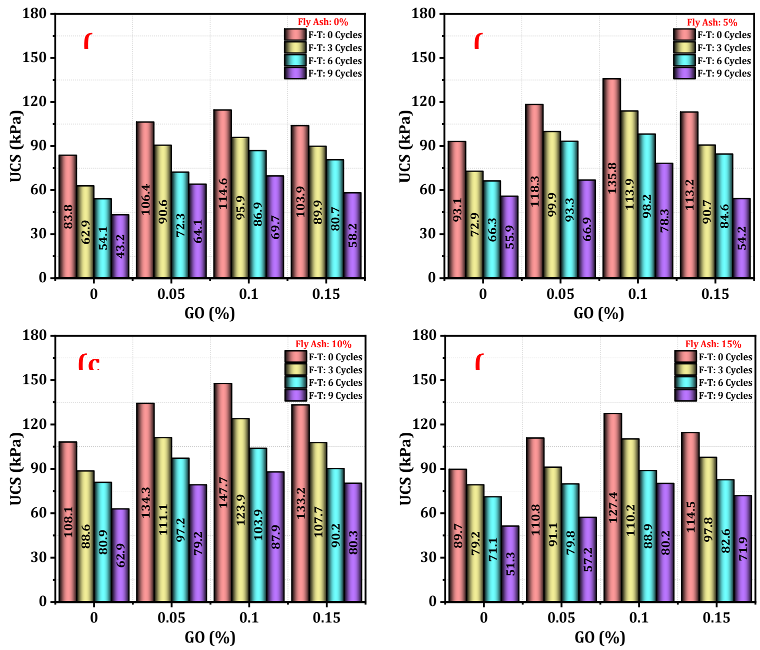

Fig. 3. Variations in UCS of expansive clay containing different percentages of GO under various F-T cycles, for: (a) 0% fly ash, (b) 5% fly ash, (c) 10% fly ash, and (d) 15% fly ash

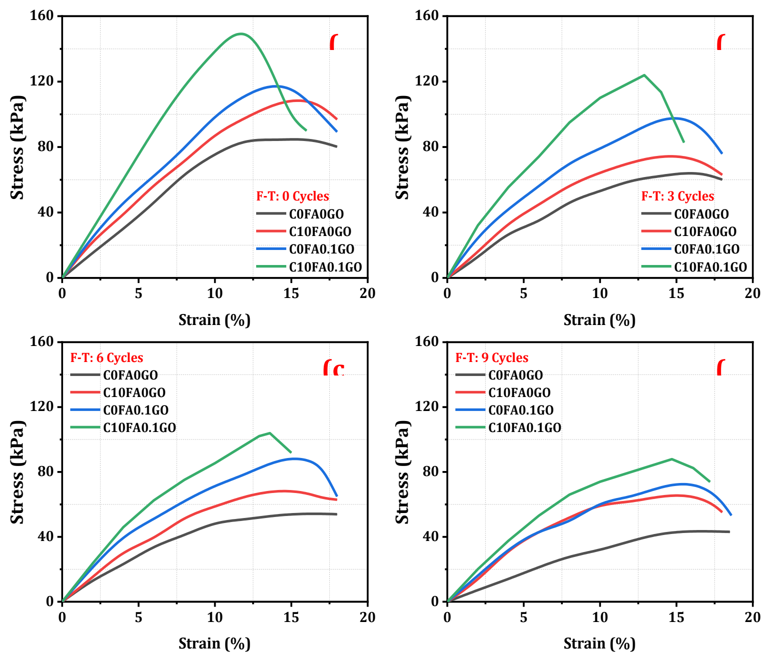

Fig. 4. Effect of the number of freeze–thaw cycles on the stress–strain behavior of the optimized samples: (a) 0 cycles, (b) 3 cycles, (c) 6 cycles, (d) 9 cycles

become softer. The reduction in peak stress and the relative increase in failure strain in subsequent cycles indicate that the soil has shifted from a quasi-brittle state toward a more ductile behavior. This behavioral change can be directly attributed to the destructive effects of the freeze-thaw cycles. With each freezing event, the water in the pores expands and exerts pressure on the soil's internal walls. These repeated pressures eventually cause microcracks, deterioration of inter-particle bonds, and a reduction in microstructure cohesion. However, samples modified with graphene oxide (GO) and fly ash (FA), compared to those without additives, experience a more controlled reduction in strength while maintaining adequate ductility. These features are highly important from a geotechnical design perspective, as materials that maintain strength while allowing greater deformation exhibit more stable performance under various loadings. In summary, the addition of GO and FA not only helps increase material strength but also improves post-peak behavior by reducing the soil structure's sensitivity to freeze-thaw induced damage, an issue of great significance in construction projects in cold regions [48,51].

4.3. Effect of GO and Fly Ash on the E50 of Expansive Clay During Freeze–Thaw Cycling

Fig. 5 illustrates the variations in the initial elastic modulus (E50) of expansive clay treated with different percentages of graphene oxide (GO) and fly ash (FA) under successive freeze–thaw cycles. The E50 parameter represents the soil's initial stiffness within small strain ranges and plays a critical role in the performance assessment of geotechnical structures under service conditions, such as embankments, retaining walls, and subgrade layers. According to the results, in the absence of freeze–thaw exposure (cycle 0), the inclusion of GO significantly increases the E50 values across all levels of fly ash. This improvement can be attributed to enhanced soil compressibility, improved interparticle contact, and a reduction in effective porosity, all resulting from the structural modification induced by GO. As freeze–thaw cycles progress, a gradual reduction in E50 is observed, primarily due to the deterioration of internal bonding, the formation of microcracks, and the increased heterogeneity within the soil matrix caused by thermal stresses. Nonetheless, samples incorporating higher FA contents demonstrate better retention of stiffness. This behavior is closely linked to the formation of secondary cementitious compounds through pozzolanic reactions between fly ash and soil constituents, which contribute to structural stability and mitigate the propagation of freeze-induced cracking. Notably, in specimens with 10% and 15% FA, the E50 values remain within an acceptable range even after extended cycling, indicating a favorable structural response under repeated thermal loading. These findings highlight the significance of selecting optimal GO and FA contents in designing geotechnical materials resilient to freeze–thaw conditions. It is also worth noting that the loss in stiffness caused by cyclic freezing and thawing may, in some cases, exceed the reduction in peak strength. This underscores the greater sensitivity of elastic response characteristics to environmental degradation, especially under early-stage loading conditions [63-65].

4.4. Durability of Expansive Clay Treated with GO and FA Under Freeze–Thaw Cycles

Fig. 6 illustrates the reduction ratio of unconfined compressive strength (UCS) in various samples containing graphene oxide (GO) and fly ash (FA) subjected to successive freeze–thaw cycles. This parameter serves as a direct indicator of the mechanical durability of soils under harsh environmental conditions, particularly in cold regions. The results indicate that incorporating GO at all FA levels effectively reduces the rate of strength loss. In other words, GO contributes to stabilizing the soil structure against degradation induced by freeze–thaw actions. Its role can be attributed to enhancing interparticle bonding, reducing permeability, and limiting the propagation of

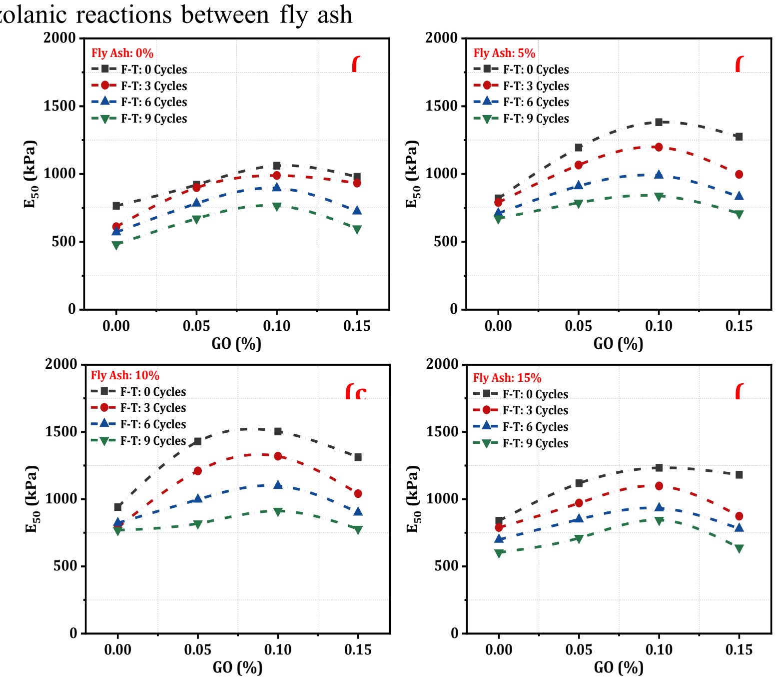

Fig. 5. Variations in E50 of expansive clay containing different percentages of GO under various F-T cycles, for: (a) 0% fly ash, (b) 5% fly ash, (c) 10% fly ash, and (d) 15% fly ash

microcracks, all of which collectively delay the onset and progression of structural deterioration. Nevertheless, the most significant impact on mitigating strength loss is associated with the presence of fly ash. Increasing the FA content, especially to 10% and 15%, leads to a considerable reduction in the UCS loss ratio. This improvement is primarily due to the pozzolanic nature of FA, which reacts with active soil components to form secondary cementitious phases. These reaction products fill pore spaces, reduce effective porosity, and ultimately enhance the structural integrity of the treated soil. Notably, in specimens with higher FA content, the rate of strength reduction becomes more gradual and stable beyond the sixth freeze–thaw cycle, suggesting a progressive structural development and stabilization of pozzolanic reactions within the soil matrix. From an engineering perspective, materials that exhibit lower strength loss under freeze–thaw cycling are more suitable for use in pavement subgrades, embankments, earth dams, and other geotechnical structures exposed to temperature fluctuations. Accordingly, a blend of GO with 10% to 15% fly ash is recommended as an effective strategy for enhancing the cyclic durability of soils in cold and frost-prone environments [66-68].

5. Conclusion

This study evaluated the mechanical performance and freeze–thaw durability of expansive clay stabilized with varying contents of graphene oxide (GO) and fly ash (FA). A series of laboratory tests, including unconfined compressive strength (UCS), stress–strain behavior, and elastic modulus (E50), were conducted on samples subjected to 0, 3, 6, and 9 freeze–thaw cycles. The analysis aimed to understand how these additives influence strength development, stiffness retention, and long-term degradation under cyclic thermal loading. Based on the experimental findings, the following conclusions can be drawn:

- The combined use of graphene oxide and fly ash significantly enhanced the unconfined compressive strength of expansive clay. The most effective performance was observed in the sample containing 10% FA and 0.1% GO, which showed approximately 76% higher UCS compared to untreated soil.
- GO contributed to improved interparticle bonding and microstructural stability, while FA provided long-term benefits through pozzolanic reactions and secondary cementation.
- As the number of freeze–thaw cycles increased, all samples exhibited reduced peak strength and increased failure strain, indicating a transition from brittle to more ductile behavior. However, treated samples showed more stable post-peak performance and better deformation capacity.
- The E50 values showed initial improvement due to GO and FA addition, with better stiffness retention in samples containing 10% and 15% FA even after repeated cycles. This highlights the role of FA in enhancing the soil's resistance to structural degradation.

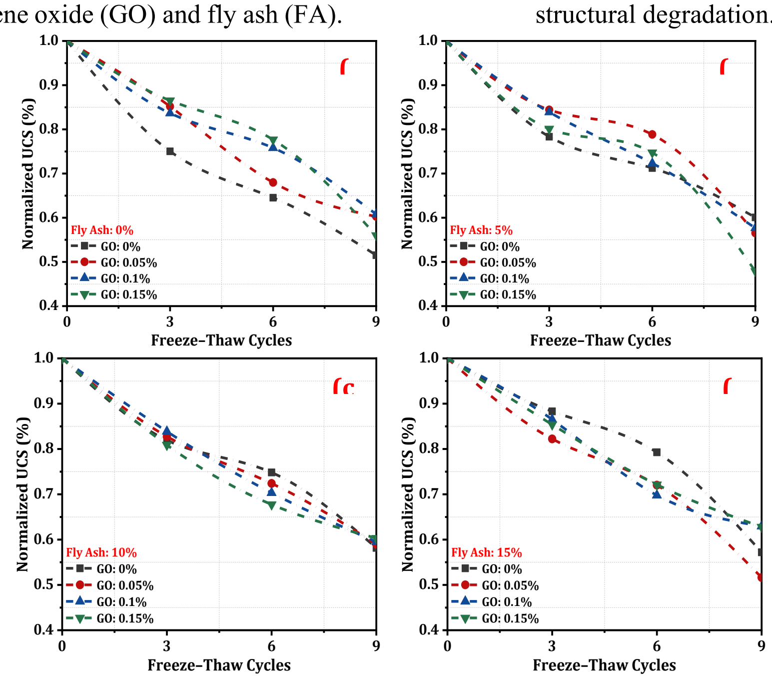

Fig. 6. Reduction ratio of unconfined compressive strength in samples containing various percentages of graphene oxide under different freeze–thaw cycles for: (a) 0% fly ash, (b) 5% fly ash, (c) 10% fly ash, and (d) 15% fly ash

- The reduction in elastic stiffness under freeze–thaw conditions was in some cases more pronounced than the loss in strength, underscoring the importance of evaluating both parameters in durability assessments.
- Durability, as measured by the UCS loss ratio, was significantly improved in samples with higher FA content. Beyond six freeze–thaw cycles, the rate of strength loss became more gradual in these mixtures, indicating stabilization of the internal soil structure.
- Overall, the use of GO in combination with 10–15% FA is recommended as a practical and effective solution for enhancing both the mechanical performance and long-term freeze–thaw durability of expansive soils in geotechnical applications, particularly in cold regions.

References

[1] Chen, L., F. Ming, X. Zhang, X. Wei, and Y. Liu. "Comparison of the hydraulic conductivity between saturated frozen and unsaturated unfrozen soils." International Journal of Heat and Mass Transfer 165 (2021): 120718. https://doi.org/10.1016/j.ijheatmasstransfer.2020.120718

[2] Sun, L., X. Chang, X. Yu, G. Jia, L. Chen, Y. Wang, and Z. Liu. "Effect of freeze-thaw processes on soil water transport of farmland in a semi-arid area." Agricultural Water Management 252 (2021): 106876. https://doi.org/10.1016/j.agwat.2021.106876

[3] Wang, H. X., Z. Z. Wu, Y. Z. Tan, X. Z. Cui, Q. J. Zuo, L. H. Wang, and L. Q. Lu. "Characteristics of pore structure of stabilized/solidified sediments during freeze–thaw cycles." Construction and Building Materials 259 (2020): 119804. https://doi.org/10.1016/j.conbuildmat.2020.119804

[4] Shastri, A., M. Sánchez, X. Gai, M. Y. Lee, and T. Dewers. "Mechanical behavior of frozen soils: Experimental investigation and numerical modeling." Computers and Geotechnics 138 (2021): 104361. https://doi.org/10.1016/j.compgeo.2021.104361

[5] Lin, J., W. Zou, Z. Han, Z. Zhang, and X. Wang. "Structural, volumetric and water retention behaviors of a compacted clay upon saline intrusion and freeze-thaw cycles." Journal of Rock Mechanics and Geotechnical Engineering 14.3 (2022): 953-966. https://doi.org/10.1016/j.jrmge.2021.12.012

[6] Qi, J., P. A. Vermeer, and G. Cheng. "A review of the influence of freeze-thaw cycles on soil geotechnical properties." Permafrost and Periglacial Processes 17.3 (2006): 245-252. https://doi.org/10.1002/ppp.559

[7] Wu, G., Y. Xie, J. Wei, and X. Yue. "Freeze-thaw erosion mechanism and preventive actions of highway subgrade soil in an alpine meadow on the Qinghai-Tibet Plateau." Engineering Failure Analysis 143 (2023): 106933. https://doi.org/10.1016/j.engfailanal.2022.106933

[8] Ng, C. W. W., Z. Li, Q. Zhang, S. Zhang, and Y. Wang. "Effects of soil structure on cyclic freeze-thaw induced volumetric behavior using a modified double-cell triaxial system." Cold Regions Science and Technology 203 (2022): 103648. https://doi.org/10.1016/j.coldregions.2022.103648

[9] Leuther, F. and S. Schlüter. "Impact of freeze–thaw cycles on soil structure and soil hydraulic properties." Soil 7.1 (2021): 179-191. https://doi.org/10.5194/soil-7-179-2021

[10] Barman, D. and S. K. Dash. "Stabilization of expansive soils using chemical additives: A review." Journal of Rock Mechanics and Geotechnical Engineering 14.4 (2022): 1319-1342. https://doi.org/10.1016/j.jrmge.2022.02.011

[11] Ikeagwuani, C. C. and D. C. Nwonu. "Emerging trends in expansive soil stabilisation: A review." Journal of Rock Mechanics and Geotechnical Engineering 11.2 (2019): 423-440. https://doi.org/10.1016/j.jrmge.2018.08.013

[12] Zada, U., A. Jamal, M. Iqbal, S. M. Eldin, M. Almoshaogeh, S. R. Bekkouche, and S. Almuaythir. "Recent advances in expansive soil stabilization using admixtures: current challenges and opportunities." Case Studies in Construction Materials 18 (2023): e01985. https://doi.org/10.1016/j.cscm.2023.e01985

[13] Shaheen, S. M., P. S. Hooda, and C. D. Tsadilas. "Opportunities and challenges in the use of coal fly ash for soil improvements–a review." Journal of Environmental Management 145 (2014): 249-267. https://doi.org/10.1016/j.jenvman.2014.07.005

[14] Noaman, M. F., M. A. Khan, K. Ali, and A. Hassan. "A review on the effect of fly ash on the geotechnical properties and stability of soil." Cleaner Materials 6 (2022): 100151. https://doi.org/10.1016/j.clema.2022.100151

[15] Ahmad, S., M. S. A. Ghazi, M. Syed, and M. A. Al-Osta. "Utilization of fly ash with and without secondary additives for stabilizing expansive soils: A review." Results in Engineering (2024): 102079. https://doi.org/10.1016/j.rineng.2024.102079

[16] Alterary, S. S. and N. H. Marei. "Fly ash properties, characterization, and applications: A review." Journal of King Saud University-Science 33.6 (2021): 101536. https://doi.org/10.1016/j.jksus.2021.101536

[17] Zimar, Z., D. Robert, A. Zhou, F. Giustozzi, S. Setunge, and J. Kodikara. "Application of coal fly ash in pavement subgrade stabilisation: A review." Journal of Environmental Management 312 (2022): 114926. https://doi.org/10.1016/j.jenvman.2022.114926

[18] Khodabandeh, M. A., G. Nagy, and Á. Török. "Stabilization of collapsible soils with nanomaterials, fibers, polymers, industrial waste, and microbes: Current trends." Construction and Building Materials 368 (2023): 130463. https://doi.org/10.1016/j.conbuildmat.2023.130463

[19] Ghasabkolaei, N., A. J. Choobbasti, N. Roshan, and S. E. Ghasemi. "Geotechnical properties of the soils modified with nanomaterials: A comprehensive review." Archives of Civil and Mechanical Engineering 17.3 (2017): 639-650. https://doi.org/10.1016/j.acme.2017.01.010

[20] Chaudhary, V., J. S. Yadav, and R. K. Dutta. "Durability performance of soil stabilized with nano additive's: a review." Indian Geotechnical Journal (2024): 1-13. https://doi.org/10.1007/s40098-024-00906-9

[21] Mathur, R. B., B. P. Singh, and S. Pande. "Carbon nanomaterials: synthesis, structure, properties and applications." Taylor & Francis (2016). https://doi.org/10.1201/9781315371849

[22] Taha, M. R., J. M. Alsharef, T. A. Khan, M. Aziz, and M. Gaber. "Compressive and tensile strength enhancement of soft soils using nanocarbons." Geomechanics and Engineering 16.5 (2018): 559-567. https://doi.org/10.12989/gae.2018.16.5.559

[23] Al-Mansob, R. A., L. Y. Jia, J. Alsharef, T. M. Jassam, J. L. Ng, S. I. A. Ali, and S. B. Surol. "Effects of carbon-nanotube and lime on the weak soil stability." In AIP Conference Proceedings 2401.1 (2021). AIP Publishing. https://doi.org/10.1063/5.0073028

[24] Kumar, A. and S. Sinha. "Support vector machine-based prediction of unconfined compressive strength of Multi-Walled Carbon nanotube doped soil-fly ash mixes." Multiscale and

Multidisciplinary Modeling, Experiments and Design 7.6 (2024): 5365-5386. https://doi.org/10.1007/s41939-024-00524-1

[25] Kishore, K., M. N. Sheikh, and M. N. Hadi. "Doped multi-walled carbon nanotubes and nanoclay based-geopolymer concrete: An overview of current knowledge and future research challenges." Cement and Concrete Composites (2024): 105774. https://doi.org/10.1016/j.cemconcomp.2024.105774

[26] Hansora, D. P., N. G. Shimpi, and S. Mishra. "Graphite to graphene via graphene oxide: an overview on synthesis, properties, and applications." Jom 67.12 (2015): 2855-2868. https://doi.org/10.1007/s11837-015-1522-5

[27] Sun, L. "Structure and synthesis of graphene oxide." Chinese Journal of Chemical Engineering 27.10 (2019): 2251-2260. https://doi.org/10.1016/j.cjche.2019.05.003

[28] Akarsh, P. K., D. Shrinidhi, S. Marathe, and A. K. Bhat. "Graphene oxide as nano-material in developing sustainable concrete–A brief review." Materials Today: Proceedings 60 (2022): 234-246. https://doi.org/10.1016/j.matpr.2021.12.510

[29] Hu, Z. Y., Y. Wan, Y. J. Duan, Y. H. Shi, C. P. Gu, R. Ma, and D. Cui. "A Review of the Impact of Graphene Oxide on Cement Composites." Nanomaterials 15.3 (2025): 216. https://doi.org/10.3390/nano15030216

[30] Huang, C. Y., Y. C. Lin, J. H. Chung, H. Y. Chiu, N. L. Yeh, S. J. Chang, and G. Y. Chen. "Enhancing cementitious composites with functionalized graphene oxide-based materials: surface chemistry and mechanisms." International Journal of Molecular Sciences 24.13 (2023): 10461. https://doi.org/10.3390/ijms241310461

[31] Alex, A. G., A. Kedir, and T. G. Tewele. "Review on effects of graphene oxide on mechanical and microstructure of cement-based materials." Construction and Building Materials 360 (2022): 129609. https://doi.org/10.1016/j.conbuildmat.2022.129609

[32] Liu, C., X. Huang, Y. Y. Wu, X. Deng, J. Liu, Z. Zheng, and D. Hui. "Review on the research progress of cement-based and geopolymer materials modified by graphene and graphene oxide." Nanotechnology Reviews 9.1 (2020): 155-169. https://doi.org/10.1515/ntrev-2020-0014

[33] Zhang, C., W. Wang, Z. Zhu, L. Shao, Y. Wan, and Y. Zhang. "Mechanical and microscopic properties of cement composite expansive soil with graphene oxide: ecofriendly modification material." International Journal of Geomechanics 23.6 (2023): 04023071. https://doi.org/10.1061/IJGNAI.GMENG-8574

[34] Sedaghat, A., M. K. Ram, A. Zayed, R. Kamal, and N. Shanahan. "Investigation of physical properties of graphene-cement composite for structural applications." Open Journal of Composite Materials 4.1 (2014): 12-21. https://doi.org/10.4236/ojcm.2014.41002

[35] Prabakar, J., N. Dendorkar, and R. K. Morchhale. "Influence of fly ash on strength behavior of typical soils." Construction and Building Materials 18.4 (2004): 263-267. https://doi.org/10.1016/j.conbuildmat.2003.11.003

[36] Kolias, S., V. Kasselouri-Rigopoulou, and A. Karahalios. "Stabilisation of clayey soils with high calcium fly ash and cement." Cement and Concrete Composites 27.2 (2005): 301-313. https://doi.org/10.1016/j.cemconcomp.2004.02.019

[37] Sezer, A., G. İnan, H. R. Yılmaz, and K. Ramyar. "Utilization of a very high lime fly ash for improvement of Izmir clay." Building and Environment 41.2 (2006): 150-155. https://doi.org/10.1016/j.buildenv.2004.12.009

[38] Lin, D. F., K. L. Lin, and H. L. Luo. "A comparison between sludge ash and fly ash on the improvement in soft soil." Journal of the Air & Waste Management Association 57.1 (2007): 59-64. https://doi.org/10.1080/10473289.2007.10465294

[39] Sharma, N. K., S. K. Swain, and U. C. Sahoo. "Stabilization of a clayey soil with fly ash and lime: a micro level investigation." Geotechnical and Geological Engineering 30.5 (2012): 1197-1205. https://doi.org/10.1007/s10706-012-9532-3

[40] Saafi, M., L. Tang, J. Fung, M. Rahman, and J. Liggat. "Enhanced properties of graphene/fly ash geopolymeric composite cement." Cement and Concrete Research 67 (2015): 292-299. https://doi.org/10.1016/j.cemconres.2014.08.011

[41] Ozdemir, M. A. "Improvement in bearing capacity of a soft soil by addition of fly ash." Procedia Engineering 143 (2016): 498-505. https://doi.org/10.1016/j.proeng.2016.06.063

[42] Naseri, F., M. Irani, and M. Dehkhodarajabi. "Effect of graphene oxide nanosheets on the geotechnical properties of cemented silty soil." Archives of Civil and Mechanical Engineering 16.4 (2016): 695-701. https://doi.org/10.1016/j.acme.2016.04.008

[43] Zhou, G. X., J. Zhong, H. Zhang, X. Hu, J. Wu, N. Koratkar, and X. Shi. "Influence of releasing graphene oxide into a clayey sand: physical and mechanical properties." RSC Advances 7.29 (2017): 18060-18067. https://doi.org/10.1039/C7RA01539A

[44] Li, M., C. Fang, S. Kawasaki, and V. Achal. "Fly ash incorporated with biocement to improve strength of expansive soil." Scientific Reports 8.1 (2018): 2565. https://doi.org/10.1038/s41598-018-20921-0

[45] Zhang, J., P. Su, K. Wen, Y. Li, and L. Li. "Mechanical performance and environmental effect of coal fly ash on MICP-induced soil improvement." KSCE Journal of Civil Engineering 24.10 (2020): 3189-3201. https://doi.org/10.1007/s12205-020-1931-z

[46] Li, D., P. Lei, H. Zhang, J. Liu, and W. Lu. "Co-Effects of Graphene Oxide and Cement on Geotechnical Properties of Loess." Advances in Materials Science and Engineering 2021 (2021): 7429310. https://doi.org/10.1155/2021/7429310

[47] Fattah, M. Y., S. A. Al-Haddad, A. A. Shahoobe, and K. R. Al-Khafaji. "Characteristics of soft clays enhanced by graphene oxide." In IOP Conference Series: Earth and Environmental Science 856.1 (2021): 012018. IOP Publishing. https://doi.org/10.1088/1755-1315/856/1/012018

[48] Aziz, M., M. Hamza, A. M. Rasool, U. Ali, T. Ahmed, Z. N. Kharal, and Z. U. Rehman. "Use of graphene oxide nanomaterial to improve mechanical properties of cement-treated silty soil." Arabian Journal for Science and Engineering 48.4 (2023): 5603-5618. https://doi.org/10.1007/s13369-022-07530-w

[49] Mohamed, A. A. M. S., J. Yuan, M. Al-Ajamee, Y. Dong, Y. Ren, and T. Hakuzweyezu. "Improvement of expansive soil characteristics stabilized with sawdust ash, high calcium fly ash and cement." Case Studies in Construction Materials 18 (2023): e01894. https://doi.org/10.1016/j.cscm.2023.e01894

[50] Zhang, J., J. Zhang, P. Miao, and Y. Shu. "Experimental investigation and analysis of the impact of salinity erosion on the strength of graphene oxide cement soil." Case Studies in Construction Materials 21 (2024): e03413. https://doi.org/10.1016/j.cscm.2024.e03413

[51] Jiajia, F., W. Wei, F. Tianhong, W. Xudong, W. Hongxiang, and X. Mengqi. "Dynamic characteristics and microscopic mechanism of graphene oxide modified coastal soft soil under small strain." Construction and Building Materials 448 (2024): 138257. https://doi.org/10.1016/j.conbuildmat.2024.138257

[52] Zhang, J. and J. Zhang. "Experimental study and analysis of the static and dynamic mechanical properties of graphene oxide cement soil under composite salinity erosion." Construction and Building Materials 451 (2024): 138848. https://doi.org/10.1016/j.conbuildmat.2024.138848

[53] Li, Y., Z. Zhong, X. Ma, Y. Gan, and X. Kang. "High-performance superhydrophobic fly ash based geopolymers with graphene oxide reinforcement for enhanced durability and repellency." Construction and Building Materials 452 (2024): 138944. https://doi.org/10.1016/j.conbuildmat.2024.138944

[54] Li, Z. and X. Shi. "Influence of Graphene Oxide on Resistance of A Fly Ash-Based Geopolymer Paste to Cyclic Freeze-Thaw Damage and Post-Damage Carbonation." Cement and Concrete Composites (2025): 106023. https://doi.org/10.1016/j.cemconcomp.2025.106023
[55] ASTM D422-63. "Standard Test Method for Particle-Size Analysis of Soils." ASTM International, Pennsylvania, United States (2007). https://doi.org/10.1520/D0422-63R07
[56] ASTM D854-14. "Standard Test Methods for Specific Gravity of Soil Solids by Water Pycnometer." ASTM International, Pennsylvania, United States (2014). https://doi.org/10.1520/D0854-14
[57] ASTM D4318-17e1. "Standard Test Methods for Liquid Limit, Plastic Limit, and Plasticity Index of Soils." ASTM International, Pennsylvania, United States (2017). https://doi.org/10.1520/D4318-17E01
[58] ASTM D698-12e2. "Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Standard Effort." ASTM International, Pennsylvania, United States (2012). https://doi.org/10.1520/D0698-12E02
[59] ASTM D2166/D2166M-13. "Standard Test Method for Unconfined Compressive Strength of Cohesive Soil." ASTM International, Pennsylvania, United States (2013).
[60] Wang, E., T. Zhou, H. Jiang, X. Liu, and C. Wu. "Effects of carbon nanotubes treatment on engineering properties of sandy fine-grained soils subject to freeze-thaw cycles." Research in Cold and Arid Regions (2025). https://doi.org/10.1016/j.rcar.2025.03.003
[61] Cwirzen, A. and K. Habermehl-Cwirzen. "The effect of carbon nano- and microfibers on strength and residual cumulative strain of mortars subjected to freeze-thaw cycles." Journal of Advanced Concrete Technology 11.3 (2013): 80-88. https://doi.org/10.3151/jact.11.80
[62] Yuan, X., M. Dai, Y. Gao, F. Liu, and M. Zhang. "Pore morphology based on graphene oxide modified steel fibre concrete for freeze–thaw resistance." Construction and Building Materials 409 (2023): 133877. https://doi.org/10.1016/j.conbuildmat.2023.133877
[63] Saride, S. and T. T. Dutta. "Effect of fly-ash stabilization on stiffness modulus degradation of expansive clays." Journal of Materials in Civil Engineering 28.12 (2016): 4016166. https://doi.org/10.1061/(ASCE)MT.1943-5533.000167
[64] James, J., S. Vijayasimhan, and E. Eyo. "Stress-strain characteristics and mineralogy of an expansive soil stabilized using lime and phosphogypsum." Applied Sciences 13.1 (2022): 123. https://doi.org/10.3390/app13010123
[65] Ranjbar, N., M. Mehrali, M. Mehrali, U. J. Alengaram, and M. Z. Jumaat. "Graphene nanoplatelet-fly ash based geopolymer composites." Cement and Concrete Research 76 (2015): 222-231. https://doi.org/10.1016/j.cemconres.2015.06.003
[66] Hotineanu, A., M. Bouasker, A. Aldaood, and M. Al-Mukhtar. "Effect of freeze–thaw cycling on the mechanical properties of lime-stabilized expansive clays." Cold Regions Science and Technology 119 (2015): 151-157. https://doi.org/10.1016/j.coldregions.2015.08.008
[67] Fakhrabadi, A., A. J. Choobbasti, and S. S. Kutanaei. "Durability evaluation of clayey sandy soil stabilized with copper-slag-based geopolymer under freezing–thawing cycles." International Journal of Pavement Research and Technology 18.1 (2025): 256-273. https://doi.org/10.1007/s42947-023-00341-8
[68] Tiwari, N., N. Satyam, and A. J. Puppala. "Strength and durability assessment of expansive soil stabilized with recycled ash and natural fibers." Transportation Geotechnics 29 (2021): 100556. https://doi.org/10.1016/j.trgeo.2021.100556