Original Article

Freeze-thaw resistance of eco-material stabilized loess

LI Guo-yu ^{1, 2, 3, 4} ^(D) https://orcid.org/0000-0002-4651-6251; e-mail: guoyuli@lzb.ac.cn

HOU Xin ⁵ https://orcid.org/0000-0001-6092-9805; e-mail: 286693463@qq.com

ZHOU Yu ^{1, 2, 4*} ^(D) https://orcid.org/0000-0003-4813-158X; ^[M] e-mail: zhouyu@lzb.ac.cn

MA Wei 1, 2, 4 Dhttps://orcid.org/0000-0001-6559-5429; e-mail: mawei@lzb.ac.cn

MU Yan-hu^{1,2,4*} ^(D) https://orcid.org/0000-0001-6553-1126; ^[M] e-mail: muyanhu@lzb.ac.cn

CHEN Dun ^{1, 2, 4} https://orcid.org/0000-0002-5357-2681; e-mail: chendun@lzb.ac.cn

TANG Li-yun ³ ^bhttps://orcid.org/0000-0002-0890-5432; e-mail: tangly@xust.edu.cn

*Corresponding authors

- 1 Da Xing'anling Observation and Research Station of Frozen–Ground Engineering and Environment, Northwest Institute of Eco-Environment and Resources, Chinese Academy of Sciences, Jiagedaqi 165000, Heilongjiang, China
- 2 State Key Laboratory of Frozen Soil Engineering, Northwest Institute of Eco-Environment and Resources, Chinese Academy of Sciences, Lanzhou 730000, China

3 College of Architecture and Civil Engineering, Xi'an University of Science and Technology, Xi'an 710054, China

4 University of Chinese Academy of Sciences, Beijing 100049, China

5 Yuexiu Property Company Limited, Guangzhou 510623, China

Citation: Li GY, Hou X, Zhou Y, et al. (2021) Freeze–thaw resistance of eco–material stabilized loess. Journal of Mountain Science 18(3). https://doi.org/10.1007/s11629-020-6308-8

© Science Press, Institute of Mountain Hazards and Environment, CAS and Springer-Verlag GmbH Germany, part of Springer Nature 2021

Abstract: In the Loess Plateau in Northern China, repeated freeze-thaw (FT) cycles deteriorate the strength and structure of loess as a foundation soil, resulting in the instability or failure of supporting structure. Lignosulfonate is an eco-material, utilized as an effective and nontraditional stabilizer to improve the engineering properties of metastable soils. A series of laboratory tests, including unconfined compression tests, cyclic loadingunloading tests and scanning electron microscopy, on calcium lignosulfonate (CL)- and sodium lignosulfonate (SL)-stabilized loess were performed to investigate the stabilization effect, deterioration mechanisms of the FT cycles, and the resistance to FT cycles. Two traditional stabilizers, quicklime (QL) and sodium silicate (SS), were selected, and the

Received: 02-Jul-2020 Revised: 11-Aug-2020 Accepted: 08-Dec-2020 engineering properties of QL- and SS-stabilized loess were compared with those of CL- and SLstabilized loess. The results showed that the strength values of CL- and SL-stabilized loess specimens decreased by 34.2% and 50% respectively, after 20 FT cycles, whereas those of the traditionally SS- and QL- stabilized specimens decreased by 85.3% and 82.87%, respectively. The elastic moduli of SL- and QL-stabilized loess specimens decreased by 22.1% and 92.0%, respectively. The mean energy dissipations of nontraditionally treated specimens also decreased significantly less than those of traditionally treated specimens. Overall, the results showed CL and SL had better stabilization effects on engineering properties of loess than QL and SS, and their stabilized loess specimens exhibited stronger resistance to FT cycles. The study findings demonstrated the significant potential of lignosulfonate for extensive application in cold loess areas.

Keywords: Freeze thaw cycle; Lignosulfonate; Loess mechanics; Eco-material; Cold region engineering; Scanning electron microscopy

1 Introduction

Loess and loess-like soils cover approximately 630,000 km² in China, approximately 7% of the total land area (Sun 2005; Li et al. 2018). Only the Loess Plateau covers approximate 317,000 km² and is one of the largest areas of loess deposits on earth (Liu 1985). Because of its low cost and wide distribution, loess is widely used as filling material and foundation soil for various infrastructures, such as buildings, roads, canals and airstrips in Northern China. However, natural loess is relative sensitive to water and is loose sediment with high porosity, low density, and high collapsibility upon wetting. Natural loess is characterized by an open and metastable structure with macropores and low interparticle bonds, and thus, exhibits poor engineering properties (Jiang et al. 2014). However, its engineering properties can be improved through physical and chemical treatment, and it is extensively used as a foundation soil.

Existing physical methods of loess improvement can be divided into three categories, 1) minimizing its sensitivity to water, 2) increasing loess compactness, and 3) replacing collapsible loess. The first category includes prewetting, which can reduce or eliminate collapsible deformation before construction on loess, and water isolation, preventing moisture's access to the loess base (Gibbs and Bara 1967; Houston and Houston 1997; Li et al. 2011; Zhang et al. 2017). The second category includes different technical methods, such as tamping, vibratory rolling, soil piling, dynamic compaction, and vibrocompaction (Mayne et al. 1984; Fei et al. 2002; Jian et al. 2003; Tu and Zhang 2010; Zhang et al. 2010). Improving the loess compactness can increase the strength and decrease the compression deformation, hydraulic conductivity and collapsible deformations of loess specimens (El-Ehwany and Houston 1990; Pereira et al. 2005; Rao and Revanasiddappa 2006; Yang and Bai 2015). The third category includes directly excavating the collapsible loess layer and replacing it with weak or nonsusceptible materials, such as coarse aggregate (Rollins and Rogers 1995; Qian 2006).

The chemical improvement of loess is mainly achieved by forming new structural bonds between soil particles, owing to chemical reactions after the stabilizer is added. Traditional chemical additives, such as lime, cement, fly ash, silicates, or any combination of these additives, have been extensively used in practice to improve the engineering properties of collapsible loess (Croft 1967; Sokolovich and Semkin 1984; Sherwood 1993; Bell 1996; Lo and Wardani 2002; Liu et al. 2019a,b). Several studies have investigated the stabilization mechanism, optimum amount of cementing materials, performance of treated loess, and advantages and disadvantages of various chemical stabilizers (Hurley and Thornburn 1972; Zia and Fox 2000; Yang et al. 2001; Lim et al. 2002; Wang et al. 2005; Jefferson et al. 2008; Mi and Hu 2009).

Generally, the loess treated using the physical or chemical improvement techniques, as described above, can satisfy the strength and deformation requirements for infrastructure construction. However, differential settlement, structural damage, pavement cracking, and landslides still occurred in infrastructures built on loess treated using physical and/or chemical methods in Northern China after several years of operation. The main reason was that the strong weathering actions, including freeze-thaw (FT), drying-wetting and salinization-desalinization deteriorated cycles, significantly engineering properties, such as the Atterberg limit, permeability, dry density, particle size and strength (Chamberlain and Gow 1979; Viklander and Eigenbrod 2000; Shen 2004; Li et al. 2012, 2015; Wang et al. 2016). In addition, the use of some traditional chemical stabilizers causes health and environmental problems. For example, chemical additives increase the pH value of the pore solution, which decreases the strength and durability of cement and steel structures exposed to the treated soils, resulting in pollution (Sherwood 1993). The treated soil also decreases the capacity to retain water and nutrients and thus, threatens the survival of living organisms (Boardman et al. 2001; Chen and Indraratna 2014). Consequently, researchers are attempting to develop other alternative chemical methods of overcoming these health and environmental problems.

Lignosulfonates, mainly including calcium lignosulfonate (CL) and sodium lignosulfonate (SL), are produced from lignin, which is the main component of liquor by-products of the timberprocessing industries (e.g., paper mills). Lignosulfonates are ecofriendly stabilizers used for soil improvement, and they have been used extensively (Santoni et al. 2002; Karol 2003; Angenent et al. 2004; Indraratna et al. 2009; Ceylan et al. 2010; Zhang et al. 2016; Ta'negonbadi and Noorzad 2017; Yang et al. 2018). Palmer (1995) and Gopalakrishnan et al. (2012) investigated the strength and optimum amount of lignosulfonate in silty sand and clayey soil, and revealed that lignosulfonate significantly increased the unconfined compressive strength (UCS) of treated soil. Puppala and Hanchanloet (1999) examined the effect of a blend of lignin and sulfuric acid on the strength and stiffness of clayey soil and found that their combination could effectively increase the shear strength and resilient modulus. Santoni et al. (2002) and Tingle and Santoni (2003) investigated the performance of clayey soil and silty sand stabilized using different nontraditional additives, including lignosulfonates, and found that the strength of lignosulfonatestabilized soils increased significantly. Pengelly et al. (1997) performed tests on expansive subgrade soil stabilized with lignosulfonate and found that the expansion of stabilized soil was effectively controlled. Vinod et al. (2012) evaluated the erosion resistance of dispersive soil stabilized with lignosulfonate and cement, and found that lignosulfonate could effectively improve the anti-erosion ability of soil, compared to traditional stabilizers such as cement. Li et al. (2019) used CL to stabilize loess and reported that it was a new ecofriendly and effective stabilizer that deserved further application in loess engineering.

In addition, some studies were focused on the interaction mechanisms between additives and soil particles. Addo et al. (2004) found that the rearrangement of soil particles was a major reason for the increased shear strength of lignosulfonatestabilized soil. Tingle et al. (2007) reported that lignosulfonates could bind soil particles together when the soil particles were coated with a thin adhesive-like film. Indraratna et al. (2008) noted that lignosulfonate increased the erosion resistance and critical shear stress of silty sand because of its cementing property. Scanning electron microscopy (SEM) images showed that strong bonding existed between particles coated with cementing materials, and the bonds were formed because of chemical reaction of lignin with minerals in silty sand.

Although researchers have investigated the physical and mechanical properties and stabilization mechanisms of lignosulfonate-stabilized soil, further research needs to be conducted extensively. For example, how does the lignosulfonate-stabilized loess respond to FT cycles in cold loess areas? and how does its engineering properties degrade when subjected to FT cycles? In addition, the FT cycle is severe weathering process in cold loess regions in China and causes frost hazards in many structures. The FT cycle can loosen the loess structure, which is highly sensitive to water, and reduce loess strength. Therefore, these are important and urgent issues that should be addressed in designing structures constructed on collapsible loess in cold regions.

The aim of this study is to investigate the resistance of lignosulfonate-treated loess to FT cycles and the deterioration mechanisms of the FT cycle. A series of experimental laboratory tests, including FT cycles, unconfined compression (UC) and cyclic unloading-reloading (UR) tests, were conducted on loess specimens treated with nontraditional additives: CL and SL, and two traditional additives: quicklime (QL) and sodium silicate (SS). The results revealed the perfect performance of the treated loess containing CL and SL, the deterioration process and the resistance to FT cycles.

2 Material and Methods

2.1 Specimen preparation

The loess used in this study was obtained from a location near YongDeng County, Gansu province, Northwestern China (Li et al. 2018). According to the Chinese engineering geological zoning of collapsible loess (MCPRC 2004), the sampling site (36°36'34" N, 103°22'05" E) is located in an arid and collapsible loess area, where the thickness of collapsible loess varies from 10 to 15 m. The physical properties of the loess are listed in Table 1. Like collapse index (Li et al. 2015), the collapsibility coefficient of undisturbed loess varied between 0.063 and 0.108 under a load of 200 kPa (Table 1). The loess is identified as strongly collapsible loess based on the Chinese loess classification standard (MCPRC 2004; MWRPRC 1999). The main chemical components of the loess obtained through X-ray diffraction analysis are listed in the Table 2. The soluble salts were measured according to the Chinese code (MCPRC 2001) (Table 2.) The main cations were sodium and calcium, and the main anions were sulphate and chloride.

m 11		D 1	• 1				CI	
Tab	e 1	Phy	vsical	pror	perti	eso	t I	loess
			, or cour	Prop		00 0	• •	

Property	Value
Specific gravity, $G_{\rm s}$	2.70
Liquid limit, W _L (%)	26.29
Plastic limit, W_P (%)	18.24
Plasticity index, PI (%)	8.05
Maximum dry density, $ ho_{ m dmax}$ (g·cm ⁻³)	1.912
Optimum moisture content, ω_{opt} (%)	13.0
Soluble salt content, (%)	0.8
Collapsibility coefficient, δ_s	0.063-0.108
Grain size distribution (%)	Value
Sand (> 0.075 mm)	14.75
Silt (0.075–0.005 mm)	66.07
Clay (< 0.005 mm)	19.18

According to the Chinese classification standard of saline soils, this loess type belongs to the sub-sulphate saline soil group (MCPRC 2001).

The collected loess was naturally air-dried in the laboratory before it was passed through a sieve with a 2-mm opening size. Stabilizers (CL, SL, and QL) of designed amounts and distilled water were thoroughly mixed with the air-dried loess. However, SS was first dissolved in distilled water before mixing to obtain uniform specimens because SS is generally a sold particle or block. Next, water corresponding to an amount at the optimum water content of 13%, the additive, and the soil were uniformly mixed and placed in a sealed plastic container for 24 h to achieve a uniform moisture distribution. Subsequently, the mixture was poured into a cylindrical steel mold and densely compacted to a density of 1.81 g/cm³. A loess specimen with a diameter of 39.1 mm and a height of 80 mm was formed after three layers of compaction to a degree of 95%. The designed stabilizer content, defined as the ratio of the dry weight of the stabilizer to the soil in this study, was 3% according to a previous study. This showed that a 3% stabilizer content was the most economical, and its treated loess sample also satisfied the strength requirement (Hou et al. 2017).

2.2 Test method

All stabilized loess specimens were sealed in plastic wrap to prevent water loss during the FT cycle. A single FT cycle involved freezing the specimens at -15° C for 12 h (MTPRC 2005) and then thaving them

at 15°C for another 12 h in a closed system. The numbers of FT cycles were 1, 5, 10, 15, and 20. When the designed number of FT cycles was achieved, the specimens were removed from the temperaturecontrolled box. Next, the soil specimens were cured for seven days and dried to a natural minimum water content of 2%. After drying, UC and UR tests were performed to determine the strength, failure strain, energy dissipation and elastic deformation of the specimens. For replication, the above tests were carried out in triplicate. SEM micrographs of different specimens stabilized with 3% stabilizer were captured to evaluate the microstructure after curing and drying for seven days.

UC tests were performed on cylindrical specimens at a strain rate of 0.5 mm/min following test specification for highway engineering (MCPRC 2004) to determine the UCS of the stabilized soils. Uniaxial cyclic UR tests, i.e., one-dimensional and unconfined compressive tests, were performed to investigate the damage behavior of the stabilized loess used in a roadbed. First, each specimen was loaded at a vertical strain rate of 0.5 mm/min until the axial strain reached 0.5% (the first peak strain). It was then unloaded at the same strain rate until the stress dropped to o kPa and maintained for 90 s without any loading to eliminate the loading effect on the next loading. Second, the specimen was reloaded up to the second peak strain of 1% at the same strain rate. Such unloading and reloading formed a full UR cycle. This cycle was repeated for designed peak stain of 1.5%, 2.0%, and 2.5% in the following three cycles, and the soil damage was monitored.

3 Experimental Results

3.1 Effect of FT cycle on mass loss

The mass loss of loess specimens is a significant indicator that reflects the resistance of the soil sample to FT cycles. In this study, the mass loss ratio was defined as the ratio of the lost mass after the FT tests to the initial soil specimen mass. The variations in the mass loss ratio for different stabilized loess specimens after subjection to the FT cycles are shown in Fig. 1.

Table 2 Main chemical components and soluble salt content of loess

		-							
Compound	SiO ₂	CaCO ₃	Al_2O_3	CaO	Fe ₂ O ₃	MgO	K ₂ O	FeO	Total
Content (%)	51.46	12.70	10.52	7.12	2.76	2.09	1.82	1.8	
Soluble salt	Ca^{2+}	Mg^{2+}	Na+	K+	$SO_{4^{2-}}$	NO ₃ -	HCO ₃ -	Cl-	
Content (%)	0.06	0.01	0.19	0.004	0.32	0.01	0.01	0.20	0.8

The mass loss ratios of the different stabilized loess specimens increased with the increasing number of FT cycles, owing to the deteriorative effect of the FT cycles. The FT cycle significantly influenced the mass loss of loess specimens stabilized with traditional SS and QL additives, demonstrating their weaker resistance to FT cycles. The mass loss ratios of SSand QL-stabilized specimens increased by 2.70% and 3.57%, respectively, after 20 FT cycles.

The SL- and CL-stabilized loess specimens showed lower mass loss ratios, i.e., 0.9% and 1.66%, respectively, after 20 FT cycles, demonstrating their stronger resistance to FT cycles. Except for FT cycles, SS-stabilized soils also experienced salt erosion because visible salt crystals appeared on the surface of the specimens after the FT cycles (Fig. 2). According to a previous study, these salts migrate from the inside to the outside of loess specimens with water during the freezing, thawing, and drying processes and eventually accumulate and crystallize on the surface (Chen et al. 1989). Therefore, the crystallization and exfoliation of soluble salts was another reason for the mass loss in SS-stabilized soils.

3.2 Effect of FT cycle on UCS

Fig. 3 depicts the stress-strain curves of the stabilized loess specimens after exposure to different FT cycles, in which the specimens were treated with CL, SL, QL and SS. All the stabilized loess specimens exhibited similar strain-softening behavior, accompanying an increasing failure strain after the FT cycles, except for the CL-stabilized specimen, owing to the higher strength (Fig. 3a). For the SS- and QL-treated soils, their stress-strain curves changed from strong strain-softening to weak strain-softening with the increasing number of FT cycles, particularly after 10 FT cycles.

Fig. 4 shows the variation in UCS with the increasing number of FT cycles. The UCS values of the treated specimens significantly decreased with the increasing number of FT cycles, indicating the deteriorative effect of the FT cycles. Moreover, the traditional SS- and QL-treated soils showed a more significant response and weaker resistance to FT cycles than the nontraditional CL- and SL-treated soils. For example, the UCS values of SS- and QL-stabilized specimens decreased by up to 85.3% and 82.9%, respectively, from 3.94 and 3.47 MPa to 0.58 and 0.6 MPa, respectively, after 20 FT cycles. Even



Fig. 1 Variations in mean mass loss ratio with deviation after different FT cycles. FT, freeze–thaw; CL, calcium lignosulfonate; SL, sodium lignosulfonate; QL, quicklime; SS, sodium silicate.



Fig. 2 Specimens after 20 freeze–thaw (FT) cycles: a) CL-treated loess specimens; b) SL-treated loess specimens; c) SS-treated loess specimens; d) QL-treated loess specimens.

their UCS values were lower than the UCS of untreated loess specimen after 10 FT cycles, signifying the weaker resistance to FT cycles. The reasons are explained as follows. The SS-stabilized specimen had a higher salt content after SS was added, relative to the unstabilized soil. Salt crystals appeared on the surface of specimens after FT cycles (Fig. 2). The SS addition induced salt expansion and salt erosion, except for FT deterioration effect. Hence, the strength of the SS-stabilized specimen was lower than that of the unstabilized soil. For the QL-stabilized specimen, ettringite mineral, a hydrous and expansive mineral, formed when lime was added to the soil (Aldaood et al. 2014), resulting in volume increase, crack formation, and lower strength. In addition to the expansive mineral presence, FT cycles also damage the structure of QL-stabilized specimen. Thus, the strength of QLstabilized specimen was also lower than that of the



Fig. 3 Stress-strain curves for different stabilized loess after different freeze-thaw (FT) cycles.



Fig. 4 Variation in unconfined compressive strength (UCS) after different freeze–thaw (FT) cycles.

unstabilized soil. The deteriorative effect of FT cycles on the other stabilized soils has also investigated in a previous study (Wang et al. 2018). The UCS values of the SL- and CL-stabilized specimens also decreased with the increasing number of FT cycles, but they exceeded those of the QL- and SS-stabilized soils; thus, SL- and CL-stabilized specimens exhibited stronger resistance to FT cycles. For instance, the UCS values of SL- and CL-stabilized specimens decreased by 34.2% and 50%, respectively, from 3.57 and 7.30 MPa to 2.35 and 3.64 MPa, respectively, after 20 FT cycles. Based on the strength values, the CL- and SL-stabilized specimens exhibited better resistance to FT cycles than the QL- and SS-stabilized specimens. These results indicate that lignosulfonate and lignin can stabilize loess in cold loess regions subjected to frequent severe FT weathering.

Fig. 5 shows the variation in the failure strain of specimens stabilized with different stabilizers after exposure to different numbers of FT cycles. The failure strain gradually increased with the increasing number of FT cycles, except for the CL-stabilized and unstabilized loess specimens, whose failure strains slightly decreased because of their higher stiffnesses. In addition, the failure strains of stabilized loess specimens were larger than those of unstabilized specimens, which indicated that FT cycles made the stabilized soil specimens relatively flexible and minimally influenced the unstabilized soil.

Based on the experimental results, most of the stress-strain curves within the 0.5% strain were linear and elastic during the initial phase. Hence, the elastic moduli were calculated at 0.5% strain in this study. Fig. 6 shows the variations in the loess specimens stabilized using different stabilizers after subjection to different numbers of FT cycles. Overall,



Fig. 5 Variation in failure strain after different freezethaw (FT) cycles for specimens stabilized by different stabilizers.

the elastic moduli of the stabilized loess specimens gradually decreased with the increasing number of FT cycles, demonstrating the deteriorative effect of the cvcle. However, the elastic moduli FT of nontraditionally stabilized specimens were higher than those of traditionally stabilized specimens. These confirmed the superior resistance of results nontraditional CL- and SL-stabilized loess specimens to FT cycles than traditional SS- and QL-stabilized specimens, similar to the strength variation. For example, the elastic moduli of nontraditionally SLstabilized and traditionally QL-stabilized loess specimens decreased by 22.1% (from 22.6 to 17.7 MPa) and 92.0% (from 17.44 to 1.4 MPa), respectively, after 20 FT cycles.

3.3 Effect of FT cycle on energy dissipation

Cyclic loading and unloading can generally result in the change in the internal energy of soils, including energy accumulation, release, dissipation, and finally, cause internal damage. However, the damage mechanisms of CL-, SL-, SS- and QL-stabilized specimens are different. It was assumed that the internal heat of the soil specimens was not exchanged with the external environment during loading and unloading. The accumulated energy due to loading in the soil specimens could be released through unloading in the elastic strain and be dissipated in the plastic strain. The energy dissipation was determined by calculating the hysteresis loop area, which reflected the degree of damage.

Fig. 7 depicts the stress-strain curves of loess



Fig. 6 Variation in elastic modulus after different freeze-thaw (FT) cycles for specimens stabilized with different stabilizers.

specimens stabilized using different stabilizers during loading and unloading after five and 20 FT cycles. Fig. 8 shows the variation in the UCS of the stabilized loess with the increasing number of FT cycles evaluated through UR tests. Fig. 9 shows a comparison of the UCS determined using different test methods, i.e., UC and UR tests. The strength values determined using the two test methods were very close, showing that the UR cycles did not change the strength properties of the specimens. The strength values decreased with the increasing number of FT cycles, demonstrating the deteriorative effect of the FT cycles. The nontraditionally stabilized loess specimens exhibited higher strength than specimens stabilized using traditional additives. In addition, the failure strain increased with the increasing number of FT cycles.

The mean energy dissipation, before the peak stress was attained, was selected as an index to assess their variations to investigate the internal damage caused by the FT cycles. Fig. 10 shows the variation in the mean energy dissipation before failure for different stabilized loess specimens with the increasing number of FT cycles. It was observed that the mean energy dissipation decreased with the increasing number of FT cycles for all treated loess specimens. The mean energy dissipation values of the CL- and SL-stabilized soils were higher than those of the soils treated with traditional SS and QL additives under the FT cycles. However, the CL- and SLstabilized specimens showed lower relative reductions in energy dissipation, implying that they retained higher energy and strength, although they had higher absolute energy dissipation because of initial higher



Fig. 7 Unloading–reloading curves of different stabilized loess specimens after exposure to different freeze–thaw (FT) cycles: (a) CL-stabilized loess; (b) SL-stabilized loess; (c) SS-stabilized loess; (d) QL-stabilized loess.



Fig. 8 Variation in unconfined compressive strength (UCS) of stabilized loess with number of freeze-thaw (FT) cycle determined through the unloading-reloading (UR) tests.

values. For example, the energy dissipation values of the CL- and SL-stabilized soils decreased by 70.5% (from 396.42 to 108.89 J/m³) and 39.87% (from 121.54 to 73.08 J/m³), respectively, after 20 FT cycles, but those of the SS- and QL-stabilized soils decreased by 91.3% (from 143.96 to 12.49 J/m³) and 81.2% (from 112.97 to 21.22 J/m³).

The elastic strain is the difference between the unloading strain at which the soil begins to be unloaded and the zero-stress strain at which the loading decreases to zero, and it represents the recoverable deformation of the soil during unloading.



Fig. 9 Comparison of unconfined compressive strength (UCS) of stabilized loess based on unloading–reloading (UR) and unconfined compression (UC) tests.

Fig. 11 shows the elastic strains of the stabilized loess specimens under different FT cycles for cyclic UR tests. Considering that most of the loess specimens were damaged after subjection to three UR cycles, only the elastic strains for the first three UR cycles were adopted for analysis in this study (Fig. 11). The elastic strain decreased with the increasing number of FT cycles. The QL- and SS-stabilized specimens showed a more significant response than the CL- and SL-stabilized specimens and their elastic strains were lower, indicating a slower recoverable capacity. The elastic strains of the QL- and SS-stabilized specimens



Fig. 10 Variation in mean energy dissipation before failure for different stabilized loess specimens after different freeze–thaw (FT) cycles.



Fig. 11 Variations in mean elastic strain after different freeze–thaw (FT) cycles.

decreased by 42.8% and 40.2%, respectively, from 1.73% and 1.69% to 0.99% and 1.01%. However, the elastic strains of the CL- and SL-stabilized specimens did not show a significant trend.

3.4 Stabilization effect on microstructure

In this study, SEM micrographs captured at ×400 and ×1000 magnification levels were used to analyze the stabilization effect of different additives at a microscopic scale (Fig. 12). It was observed that untreated loess specimens had an openwork structure and fabrics with aggregated grain cluster and face-to-face or edge-to-face contacts. The SS-stabilized loess specimens had a thin layer of colloids on the particles. The QL-stabilized loess specimens had more column-shaped particles, i.e., the newly created calcium carbonate, which filled the voids and densified the loess. The CL- and SL-stabilized loess specimens had



Fig. 12 Scanning electron microscopy (SEM) micrographs of soil specimens treated with different stabilizers at ×400 and ×1000 magnifications, respectively; (a) and (b) unstabilized specimens, (c) and (d) 3% CL-stabilized specimens, (e) and (f) 3% SL-stabilized specimens, (g) and (h) 3% SS-stabilized specimens, (i) and (j) 3% QL-stabilized specimens.

numerous flocculated structures and irregularly aggregated particles that filled the voids and covered the particles because of newly created organic molecules. These molecules increased the density of the loess and strengthened the interparticle adhesion.

4 Discussion

Previous studies have shown that soils treated with traditional stabilizers, such as QL, SS, lime, fly ash, and cement, exhibited strong geotechnical properties. Soils in cold regions are frequently subjected to severe FT weathering, and their engineering properties can deteriorate gradually. Researchers have developed better and ecofriendly stabilizers, such as lignosulfonates, to improve the engineering properties of loess soil (Li et al. 2019). Lignosulfonate has also been used to stabilize clayey soils and sandy silt and exhibited a similar enhancement effect on strength (Chen and Indraratna 2014; Ta'negonbadi and Noorzad 2017; Alazigha et al. 2018). However, the resistance of lignosulfonate-stabilized loess to FT cycles has not been reported in the literature. In this study, the resistance of lignosulfonate-stabilized loess to the FT cycle was investigated and compared with those of specimens treated with the conventional QL- and SS-stabilizers, and the deteriorative effect of the FT cycle was revealed. The findings of this study demonstrate that FT cycles can significantly deteriorate the geotechnical properties of stabilized loess specimens. However, the loess specimens stabilized using the nontraditional CL and SL stabilizers exhibited stronger resistance to FT cycles than QL- and SS-stabilized loess specimens.

The stabilization mechanisms of different stabilizers have been reported in the literature. The increase in the strength of QL-stabilized soil specimens is mainly attributed to pozzolanic reactions. Moreover, several FT cycles can destroy the structure of stabilized soils and decrease the resistance to FT cycles (Aldaood et al. 2014; Yıldız and Soğancı 2012). For the SSstabilized soil, a new gel-like cementing compound called sodium aluminosilicate hydrate was formed, which increased the soil strength (Brough and Atkinson 2002; Latifi et al. 2014). In addition, salt expansion caused by sodium aluminosilicate hydrate negatively affected the resistance to FT cycles.

Two primary reasons explain the deterioration mechanisms of FT cycles on geotechnical properties of soils. On the one hand, the water–ice phase transformation caused the formation of macropores and fissures under the FT cycles. Generally, the pore-water volume expands by 9% when the water–ice phase change occurs during freezing. When the ice-induced expansive force is higher than the interparticle bonding force, the relative movement of soil particles occurs. When the soil thaws, the moved particles cannot return completely (Li et al. 2017, 2018), thus result in larger pores, looser structure, and volume expansion. On the other hand, freezing caused the migration, accumulation, and crystallization of soluble salts in pore water because of the high salt content (0.8%, listed in Table 2), particularly sodium sulfate. The crystallization of soluble salts can also increase the soil volume and deteriorate the geotechnical properties of loess. Therefore, the resistance to FT cycles can be achieved by controlling the water/salt migration, which will enhance the interparticle bonding force and decrease the soluble salt content.

Consistent with previous studies on loess, the loess components, i.e., clinochlore and gaultite, disappeared, owing to the chemical reactions during mixing with lignosulfonate (Li et al. 2019). These reactions resulted in the higher quartz, calcite, and dolomite contents in the treated loess specimens, silica which enhanced the and carbonate cementations between the particles, decreased the spacing between the mineral crystal plane and the thickness of electric double-layer of particle, improved the compactness of the structure, bonding force, and cementing property of lignosulfonate, and ultimately increased the strength of the loess structure (Shi and Kong 2011; Addo et al. 2004). In addition, the stabilization effect of lignosulfonate also contributed to the decrease in moisture susceptibility. The charge neutralization (CN) reaction occurred after the stabilizer was added. Furthermore, the soil structure changed from the initial loose structure (because of the existence of hydrophilic clay minerals before the CN reaction) to compact structure (owing to their disappearance after CN reaction), and the soil particles were coated by hydrophobic aggregates (Gow et al. 1961; He 2015). These processes minimized moisture/salt migration in the soil and mitigated frost heave and salt expansion. Based on the two reasons explained above, lignosulfonatestabilized soil, particularly specimens treated with CL, showed a stronger resistance to FT cycles. In terms of resistance to FT cycles, the SL-stabilized soils were ranked after the CL-stabilized soils because of their relatively high salt content and salt expansion.

5 Conclusion

A series of experimental tests were performed in this study to investigate the resistance of lignosulfonate-stabilized loess to FT cycles, reveal the deterioration process of geotechnical properties, and further analyze their mechanisms. The following conclusions can be drawn:

1) The FT cycle had a significant effect on mass loss, UCS, elastic modulus, energy dissipation, and elastic strain. The CL- and SL-stabilized loess specimens exhibited stronger resistance to FT cycles, owing to the enhanced bonding force between the particles and the cementing property of lignosulfonate, compared to the QL- and SS-stabilized loess specimens.

2) The UCS values of nontraditionally CL- and SL-stabilized loess specimens decreased by 34.2% and 50%, respectively, after 20 FT cycles, while those of the traditionally SS- and QL-stabilized specimens decreased by 85.3% and 82.87%, respectively. The elastic moduli of nontraditionally SL-stabilized and nontraditionally QL-stabilized loess specimens decreased by 22.1% and 92.0%, respectively, after 20 FT cycles.

3) The mean energy dissipation values of nontraditionally CL-stabilized and traditional SSstabilized specimens decreased by 72.5% and 91.3%,

References

- Addo JQ, Sanders TG, Chenard M (2004) Road Dust Suppression: Effect on Maintenance Stability, Safety and the Environment,
- Phases 1-3. MPC Report No. 04-156. Alazigha DP, Indraratna B, Vinod JS, et al. (2018) Mechanisms of stabilization of expansive soil with lignosulfonate admixture. Transp Geotech 14:81-92.
- https://doi.org/10.1016/j.trgeo.2017.11.001 Aldaood A, Bouasker M, Al-Mukhtar M (2014) Impact of freezethaw cycles on mechanical behaviour of lime stabilized gypseous soils. Cold Reg Sci Technol 99: 38-45.
- http://doi.org/10.1016/j.coldregions.2013.12.003 Angenent LT, Karim K, Aldahhan MH, et al. (2004) Production of bioenergy and biochemicals from industrial and agricultural wastewater. Trends Biotechnol 22: 477-485.
- https://doi.org/10.1016/j.tibtech.2004.07.001 Bell FG (1996) Lime stabilization of clay minerals and soils. Eng Geol 42: 223-237. https://doi.org/10.1016/0013-7952(96)0002
- Boardman DI, Glendinning S, Rogers CF, et al. (2001) In situ monitoring of lime-stabilized road subgrade. Transp Res Rec: J Transp Res Board 1757: 3-13. https://doi.org/10.3141/
- Brough AR, Atkinson A (2002) Sodium silicate-based, alkaliactivated slag mortars: Part I. Strength, hydration and microstructure. Cement Concrete Res 32: 865-879.
- https://doi.org/10.1016/S0008-8846(02)00717-2 Ceylan H, Gopalakrishnan K, Kim S (2010) Soil stabilization with bioenergy coproduct. Transp Res Rec: J Transp Res Board 2186: 130-137. https://doi.org/10.3141/2186-14 Chamberlain EJ, Gow AJ (1979) Effect of freezing and thawing on
- the permeability and structure of soils. Eng Geol 13: 73-92. https://doi.org/10.1016/0013-7952(79)90022-X nen Q, Indraratna B (2014) Deformation
- Chen behavior lignosulfonate-treated sandy silt under cyclic loading. J Geotechl Geoenviron 141: 1-5.

- https://doi.org/10.1061/(ASCE)GT.1943-5606.0001210 Chen X, Qiu G, Wang Y, et al. (1989) Salt redistribution and heave of saline soil during cooling. J Glaciol Geocryol 11: 231-238. (In Chinese)
- Croft JB (1967) The Influence of soil mineralogical composition on cement stabilization. Geotechnique 17: 119-135. https://doi.org/10.1680/geot.1967.17.2.110
- El-Ehwany M, Houston SL (1990) Settlement and moisture movement in collapsible soils. J Geotech Eng 116: 1521-1535. https://doi.org/10.1061/(ASCE)0733-9410(1990)116:10(1521)
- Fei XZ, Wang Z, Zhou ZB (2002) Experimental research of dynamic

respectively, after 20 FT cycles. The elastic strains of the traditionally QL- and SS-stabilized specimens decreased by 42.8% and 40.2%, respectively, but the nontraditionally QL- and SS-stabilized specimens showed no significant trend.

4) The stabilized loess deterioration caused by FT cycles was attributed to the water-ice phase moisture/salt transformation, migration, and crystallization of soluble salts.

Acknowledgements

This study was funded by the China's Second Tibetan Plateau Scientific Expedition and Research (2019QZKK0905), the National Natural Science Foundation of China (Nos. 41672310 and U1703244), the Science and Technology Major Project of Gansu Province (143GKDA007) and Research Project of the State Key Laboratory of Frozen Soils Engineering (SKLFSE-ZY-20).

compaction of loess. Rock Soil Mech 23: 437-441. (In Chinese) Gopalakrishnan K, Leeuwen JV, Brown RC (2012) Sustainable Bioenergy and Bioproducts. Springer, London. Gow AJ, Davidson DT, Sheeler JB (1961) Relative effects of

- chlorides, lignosulfonates and molasses on properties of a soilaggregate mix. Highway Research Board Bulletin 282: 66-83.
- Gibbs HJ, Bara JP (1967) Stability problems of collapsing soil. J Soil
- Mech Found Div 93: 577-594. He ZQ (2015) Study on influence factors and mechanism of reubforced soil with bio-based soil stabilized. Northwest A&F
- University, Yangling, Shaanxi, China. Hou X, Ma W, Li GY, et al. (2017) Influence of ligonosulfonate on mechanical properties of Lanzhou loess. Rock Soil Mech 38: 18-26. (In Chinese)
- Hurley CH, Thornburn TH (1972) Sodium silicate stabilization of soils: A review of the literature. Highway Research Record 381: 46-79, Highway Research Board, US
- Houston S, Houston W N (1997) Collapsible soils engineering. Geotechnical Special Publication (68 ed.), Logan UT, USA, pp. 199-232
- Indraratna B, Muttuvel T, Khabbaz H, et al. (2008) Predicting the erosion rate of chemically treated soil using a process simulation apparatus for internal crack erosion. J Geotechl Geoenviron 134: 837-844

- https://doi.org/10.1061/(ASCE)1090-0241(2008)134:6(837) Indraratna B, Muttuvel T, Khabbaz H (2009) Modelling the erosion rate of chemically stabilized soil incorporating tensile force deformation characteristics. Can Geotech J 46: 57-68. https://doi.org/10.1139/T08-103
- Jefferson I, Evstatiev D, Karastanev D (2008) The treatment of collapsible loess soils using cement materials, GeoCongress 2008, American Society of Civil Engineers, New Orleans, Louisiana, the United States. https:// /doi.org/10.1061/40971(310)8
- Jiang MJ, Li T, Hu HJ, et al. (2014) DEM analyses of onedimensional compression and collapse behaviour of unsaturated structural loess. Comput Geotech 60: 47-60.

- https://doi.org/10.1016/j.compgeo.2014.04.002 Jian Z, Mincai J, Yong C (2003) Vibroflotation compaction of silty fine sands without additional backfill materials. Chin J Rock Mech Eng 22: 1350-1355. (In Chinese)
- Karol RH (2003) Chemical grouting and soil stabilization (3rd ed.), Marcel Dekker Inc., New York.
- Latifi N, Eisazadeh A, Marto A (2014) Strength behavior and microstructural characteristics of tropical laterite soil treated with

sodium silicate-based liquid stabilizer. Environ Earth Sci 72: 91-98. https://doi.org/10.1007 512665-013-2939-

- i HS, Teng WC, Zhao Q, et al. (2011) Experiment and application of prewetting method treating on collapsible loess foundation. Building Sci 27: 36-40. (In Chinese) Li GY, Ma W, Zhao SP, et al. (2012) Effect of freeze-thaw cycles on
- mechanical behavior of compacted fine-grained soil. In: Morse B, Doré G (eds.), Cold Regions Engineering 2012, American Society
- b) Colored Colore A review. Sci Cold Arid Reg 7: 456-468. J.1226
 - 6.2015.00456
- Li GY, Ma W, Mu YH, et al. (2017) Effects of freeze-thaw cycle on engineering properties of loess used as road fills in seasonally frozen ground regions, North China. J Mt Sci 14: 356-368. doi.org/10.1007/s11629-016-4005
- Li GY, Wang F, Ma W, et al. (2018) Variations in strength and deformation of compacted loess exposed to wetting-drying and freeze-thaw cycles. Cold Reg Sci Technol 151: 159-167. https://doi.org/10.1016/j.coldregions.2018.03.021 Li GY, Hou X, Mu YH, et al. (2019) Engineering properties of loess stabilized by a type of eco-material, calcium lignosulfonate. Arab
- J Geosci 12: 700. https://doi.org/10.1007/s12517-019-4876-0 Lim S, Jeon W, Lee J, et al. (2002) Engineering properties of water/wastewater-treatment sludge modified by hydrated lime, fly ash and loess. Water Res 36: 4177-4184. os://doi.org/10.1016/S0043-1354(02)00150-1

- Liu D (1985) Loess and the environment. Science Press, Beijing, China. Liu X, Cai G, Liu L, et al. (2019a). Thermo-hydro-mechanical properties of bentonite-sand-graphite-polypropylene fiber mixtures as buffer materials for a high-level radioactive waste repository. Int J Heat Mass Tran 141: 981-994.
- https://doi.org/10.1016/j.ijheatmasstransfer.2019.07.015 Liu L, Cai G, Liu X, Liu S, et al. (2019b) Evaluation of thermal-mechanical properties of quartz sand-bentonite-carbon fiber mixtures as the borehole backfilling material in ground source heat pump. Energ Buildings 202: 109407.
- https://doi.org/10.1016/j.enbuild.2019.109407 Lo SR, Wardani SP (2002) Strength and dilatancy of a silt stabilized by a cement and fly ash mixture. Can Geotech J 39: 77-89.
- https://doi.org/10.1139/t01-062. Mayne PW, Jones JS, Dumas JC (1984) Ground response to dynamic compaction. J Geotech Eng 110: 757-774. https://doi.org/10.1061/(ASCE)0733-9410(1984)110:6(757)

- Mi HZ, Hu YN (2009) Experimental Study on Engineering Property of Lime-Loess in Lanzhou. Building Sci 1: 55-58. (In Chinese) Ministry of Construction of the People's Republic of China (MCPRC)
- (2001) Code for investigation of geotechnical engineering, GB 50021-2001. Architecture and Building Press, Beijing, China. (In (hinese)
- MCPRC (2004) Code for building construction in collapsible loess regions, GB 50025-2004. Architecture and Building Press, Beijing, China. (In Chinese)
- Ministry of Transport of the People's Republic of China (MTPRC) (2005) Test Methods of Rock for Highway Engineering, JTG E41-2005. China Communications Press, Beijing, China. (In Chinese) Ministry of Water Resources of the People's Republic of China
- (MWRPRC) (1999) Specification of soil test, SL 237-1999. China Water Power Press, Beijing, China. (In Chinese) Palmer JT (1995) Strength and density modification of unpaved
- road soils due to chemical additives. Chemical Agents.
- Pengelly AD, Boehm DW, Rector E, et al. (1997) Engineering experience with in-situ modification of collapsible and expansive soils. In: Pengelly A D, Boehm DW, Rector E, Welsh J P (eds.),
- Unsaturated Soil Engineering Practice. ASCE, pp 277-298. Pereira JH, Fredlund DG, Neto MPC, et al. (2005) Hydraulic behavior of collapsible compacted gneiss soil. J Geotech Geoenviron 13: 1264-1273. https://doi.org/10.1061/(ASCE)1090-0241(2005)131:10(1264) Puppala A, Hanchanloet S (1999) Evaluation of a new chemical
- treatment method on strength and resilient properties of a cohesive soil. Transportation Res Rec 1999: 990389.
- Qian Z (2006) Main technical problems and engineering measures of railway in collapsible loess area. Chin Railway 2: 28-32. (In Chinese)
- Rao SM, Revanasiddappa K (2006) Influence of cyclic wetting drying on collapse behaviour of compacted residual soil. Geotech

Geol Eng 24: 725-734. https://doi.org/10.1007/s10706-004-5077-4 Rollins KM, Rogers GW (1995) Closure of "mitigation measures for small structures on collapsible alluvial soils". J Geotech Eng 120: 1533-1553.

- /doi.org/10.1061/(ASCE)0733-9410(10 95)121:10(7 http Santoni R, Tingle J, Webster S (2002) Stabilization of silty sand with nontraditional additives. Transport Res Recd 1787: 61-70.
- https://doi.org/10.3141/1787-07 Shen ZJ (2004) Weathering resistant design: an important aspect of
- future development of geotechnical engineering design.Chin J Geotech Eng 26: 866-869. (In Chinese)
- Sherwood PT (1993) Soil stabilization with cement and lime: state-of-the-art review. TRL State of the Art Review. Publisher: Her Majesty Stationary Office. ISBN: 9780115511714.
- Shi ZM, Kong XL (2011) Engineering Geology. 2nd Edition. China Construction Industry Press, Beijing, China. (In Chinese) Sokolovich V, Semkin V (1984) Chemical stabilization of loess soils.
- Soil Mech Found Eng 21: 149-154.
- https://doi.org/10.1007/BF01710605 Sun JZ (2005) Loessology. Hong Kong Archaeology Society, Hong Kong. (In Chinese)
- Ta'negonbadi B, Noorzad R (2017) Stabilization of clayey soil using lignosulfonate. Transp Geotech 12: 45-55. http://doi.org/10.1016/j.trgeo.2017.08.004

- http://doi.org/10.1016/j.frge0.2017.05.004 Tingle J, Santoni R (2003) Stabilization of clay soils with nontraditional additives. Transport Res Rec 1819: 72-84. https://doi.org/10.3141/1819b-10 Tingle J, Newman J, Larson S, et al. (2007) Stabilization
- mechanisms of nontraditional additives. Transport Res Rec 1989: 59-67. https://doi.org/10.3141/1989-49 Tu C, Zhang W (2010) Application of heavy tamping method in
- Yu C, Zhang W (2010) Application of nearly ampling method in foundation treatment of collapsible loss of South-to-North Water Diversion Project. Yangtze River 41: 22-24. (In Chinese)
 Viklander P, Eigenbrod D (2000) Stone movements and permeability changes in till caused by freezing and thawing. Cold Reg Sci Technol 31: 151-162.

/doi.org/10.1016/S0165-232X(00)00009-4

- Vinod JS, Mahamud MA, Indraratna B (2012) Elastic modules of soils treated with lignosulfonate. In: Narsilio G A, et al. (eds.), 11th Australia-New Zealand Conference on Geomechanics: Ground Engineering in a Changing World. Australia, Engineers Australia, pp 487-492
- Wang D, Zentar R, Abriak NE (2018) Durability and swelling of solidified/stabilized dredged marine soils with Class-F fly ash, cement, and lime. J Mater Civil Eng 30: 04018013. https:// doi.org/ 10.1061/(ASCE)MT.1943 -5533.000218
- Wang SL, Lv QF, Baaj H, et al. (2016) Volume change behaviour and microstructure of stabilized loess under cyclic freeze-thaw conditions. Can J Civil Eng 43: 865-874.

https://doi.org/10.1139/jce-2016-0052 Wang YM, Liang B, Ma XN, et al. (2005) Experimental Study of Cement loess for High-speed Railway Embankment. J Lanzhou

- Jiaotong Univ (Nat Sci) 24: 28-31. (In Chinese) Yang B, Zhang Y, Ceylan H, et al. (2018) Assessment of soils stabilized with lignin-based byproducts. Transp Geotech 17: 122-132. https://doi.org/10.1016/j.trge0.2018.10.005 Yang J, Bai X (2015) Nonlinear compression stress-strain relationship
- of compacted loess and its application to calculation of foundation
- Settlement. Rock Soil Mech 36: 1002-1008. (In Chinese)
 Yang Y, Liang B, Ding L (2001) Experimental study on the strength behaviors of flyash-lime or flyash-cement. Chin J Geotech Eng 23: 227-230. (In Čhinese)
- Yıldız M, Soğancı AS (2012) Effect of freezing and thawing on strength and permeability of lime-stabilized clays. Sci Iran 19:
- 1013-1017. https://doi.org/10.1016/j.scient.2012.06.003 Zhang QZ, Yang RF, Dai JL (2010) Analysis on compaction property of loess by vibrating roller. J Chang'an Univ (Nat Sci Ed) 30: 27-30. (In Chinese)
- Zhang T, Cai G, Liu S, et al. (2016) Engineering properties and microstructural characteristics of foundation silt stabilized by lignin-based industrial by-product. KSCE J Civ Eng 20: 2725-2736. http 10.100
- Zhang T, Cai G, Liu S (2017) Application of lignin-based by-product stabilized silty soil in highway subgrade: A field investigation. J

 Clean Prod 142: 4243-4257. https://doi.org/10.1016/j.jclepro.2016.12.002
 Zia N, Fox PJ (2000) Engineering properties of loess-fly ash mixtures for roadbase construction. Transport Res Rec 1714: 49-(1) 56. https://doi.org/10.3141/1714-07