

Mechanical and microstructural characterization of fiber-reinforced polyurethane-treated clays under freeze-thaw cycles

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ABSTRACT: Expansive clays with high plasticity are vulnerable to deterioration under repeated freeze–thaw (F–T) cycles, posing challenges for infrastructure durability in cold climates. To enhance their resilience, this study explores a sustainable stabilization–reinforcement approach using polyurethane (PU) foam as a chemical binder and waste tire textile fibers (WTTFs) as mechanical reinforcement. Laboratory investigations encompassing unconfined compressive strength (UCS) and scanning electron microscopy (SEM) were performed to examine how different WTTF contents interact with PU stabilization in improving soil resistance to F–T cycles. The results revealed that while repeated F–T cycling led to notable strength degradation in all specimens, the presence of WTTFs significantly mitigated these losses. In particular, PU-treated soils reinforced with an optimal dosage of WTTFs exhibited superior durability, retaining 15% to 40% more strength post–F–T exposure compared to unreinforced or solely PU-stabilized samples. Microstructural observations confirmed these findings, highlighting enhanced interparticle bonding, reduced microcracking, and minimized mass loss and volumetric instability in the composite specimens. These outcomes demonstrate the synergistic potential of PU and WTTFs in enhancing the long-term mechanical performance of expansive clays under harsh environmental conditions. Incorporating recycled fibrous waste into polymer-stabilized soils offers a practical, eco-friendly strategy for improving the service life of earth structures in freeze-prone regions.

KEYWORDS: Expansive clay; Freeze-Thaw (F-T) cycles; Polyurethane (PU); Waste Tire Textile Fiber (WTTF); Mechanical behavior.

1 INTRODUCTION

With the intensification of global warming, freeze–thaw (F–T) cycles have become more frequent and severe in cold regions, posing significant challenges to the geotechnical performance of soils (Ghazavi et al., 2023; Vakili et al., 2019). Fluctuations around the freezing point cause repeated expansion and contraction of water within the soil matrix, as pore water turns into ice and expands, then thaws and contracts again. This cycle generates internal stresses, leading to cracking, volumetric instability, and degradation in strength and stiffness (Ghazavi and Roustaei, 2013; Yarbaşı et al., 2007). Expansive clays are particularly vulnerable due to their high moisture sensitivity and tendency to swell, shrink, and fissure under temperature fluctuations (Alonso et al., 2005; Chaduvula et al., 2017; Xu et al., 2018). During thawing, meltwater infiltration increases pore pressure and reduces effective stress, further weakening the soil and making it susceptible to deformation and shear failure (Lu et al., 2019). These processes significantly impact the long-term performance of geotechnical structures, particularly where multiple F–T cycles occur (Roustaei et al., 2015). To mitigate these effects, stabilizing agents have been widely used to improve soil behavior under F–T conditions. Traditional binders like lime and cement have shown effectiveness in enhancing strength and durability by promoting pozzolanic reactions, reducing plasticity, and increasing resistance to water-induced volume changes (Jamsawang et al., 2017; Sharma and Sivapullaiah, 2016). However, their long-term performance under repeated F–T cycles may be compromised due to the formation of microcracks and stiffness degradation over time. Due to growing concerns over carbon emissions, resource depletion, and environmental leaching, researchers have increasingly turned to more sustainable stabilization methods that not only reduce ecological impact but also offer better adaptability to climate-related stresses (Caravaca et al., 2017; Khoshdel Sangdeh et al., 2024; Rogers et al., 2009).

Among these, polymer-based stabilizers have gained increasing attention for their chemical resistance, high adhesion, and long-term durability even under harsh environmental fluctuations, making them promising alternatives to traditional cementitious materials (Anagnostopoulos, 2015; Kolay and Dhakal, 2020; Salimi et al., 2024a).

Within the group of stabilizing agents, polyurethane (PU) stands out due to its low viscosity, rapid setting characteristics, strong adhesion to soil particles, and its ability to significantly enhance mechanical properties (Ma et al., 2023; Wang et al., 2021). Its hydrophobic nature and ability to form protective coatings around soil particles reduce water ingress and help maintain soil integrity during F–T cycling. For example, Ghasemi et al. (2023) reported improved shear strength in sandy soils with up to 10% PU, while Wang et al. (2024) observed a 42% increase in unconfined compressive strength in PU-treated permafrost melt soils. Li et al. (2025) similarly found that PU could triple the compressive strength of coral sand. These findings highlight PU's potential as a versatile stabilizer across different soil types and environmental conditions. Despite these benefits, PU-stabilized soils often exhibit brittleness and reduced performance under repeated F–T exposure due to limited ductility and strain accommodation (Fang et al., 2015).

Because PU can be brittle and vulnerable under F–T conditions, researchers have turned to fiber reinforcement as a potential solution. Fibers enhance strength and ductility through mechanisms like mechanical interlocking, friction, and energy absorption, without chemically altering the soil matrix (Gao et al., 2022; Jafari and Esna-ashari, 2012; Liu et al., 2020). Both synthetic and natural fibers have demonstrated effectiveness in controlling crack propagation and improving post-peak behavior. In recent years, waste tire textile fibers (WTTFs), a byproduct of tire recycling, have gained attention as a sustainable and cost-efficient reinforcement material

(Abbaspour et al., 2019; Narani et al., 2020). These fibers offer high tensile strength, flexibility, and environmental benefits, aligning well with circular economy principles. Several studies have confirmed the positive impact of WTTFs on soil performance. For example, Habibi et al. (2021) reported that lime-stabilized soils reinforced with WTTFs showed enhanced unconfined compressive strength (UCS), tensile strength, and toughness. Similarly, Mirzababaei et al. (2018) and Hejazi et al. (2012) found that fiber inclusion helps retain residual strength and reduce crack development, particularly under cyclic loading. Although these benefits are well recognized, the combined effects of WTTFs and PU under F–T conditions have not been sufficiently examined and require further research.

This research addresses that gap by investigating the combined effect of PU and WTTFs on the mechanical and microstructural behavior of expansive clay subjected to repeated F–T cycles. A series of laboratory tests were conducted on samples stabilized with 8% PU and reinforced with varying WTTF contents (0–4%). All specimens were exposed to eight F–T cycles and evaluated using UCS and scanning electron microscopy (SEM). Results show that the integration of WTTFs into PU-stabilized clay not only mitigates brittleness but also enhances strength, ductility, and durability during cyclic freezing and thawing. The findings support the development of a sustainable and high-performance soil stabilization strategy for cold-region infrastructure.

2 MATERIALS & METHODS

2.1 Tested materials

2.1.1 Expansive clay soil

In this study, bentonite clay was selected due to its high expansiveness and fine-grained nature, which make it especially prone to deterioration under F–T conditions. This vulnerability poses a major challenge in geotechnical applications within cold regions (Ghazavi and Roustaei, 2010). The tested material, obtained from South Khorasan, Iran, exhibited a uniform white color and soft texture. As shown in Fig. 1, its grain size distribution indicates that more than 90% of the particles are smaller than 0.075 mm, confirming its classification as a fine-grained soil. In addition, a series of geotechnical tests were conducted to evaluate its physicochemical characteristics, the results of which are summarized in Table 1.

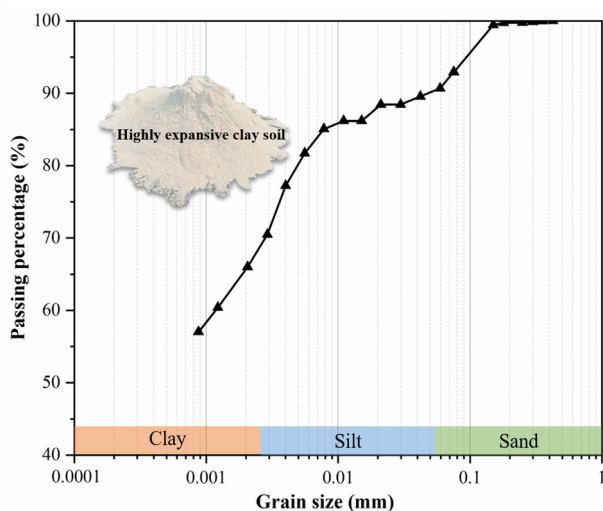


Figure 1. Grain size distribution curve of South Khorasan bentonite.

Table 1. Comprehensive characterization of the tested bentonite clay

Category	Parameter	Value / Type	Standard/Method
Geotechnical	USCS	CH	ASTM (2017) D2487
	G_s	2.56	ASTM (2014) D854
	LL (%)	294	ASTM (2017) D4318
	PL (%)	56.39	ASTM (2017) D4318
	PI (%)	237.6	ASTM (2017) D4318
	MDD (g/cm^3)	1.34	ASTM (2021) D698
	OMC (%)	32.2	ASTM (2021) D698
Chemical	pH	9.85	ASTM (2013) D4972
	EC (mS/m)	1100	ASTM (2014) D1125
Mineralogy (XRF)	SiO ₂	56.11%	X-ray Fluorescence
	Al ₂ O ₃	14.84%	
	Fe ₂ O ₃ + MgO + CaO	13.36%	
	Na ₂ O + K ₂ O	2.42%	
	LOI	11.71%	

2.1.2 Polyurethane (PU)

PU was employed as the stabilizing agent in this study due to its notable mechanical strength, low density, expansive properties, and strong adhesion to soil particles (Alinejad et al., 2019; Xiao et al., 2015). The PU polymer was synthesized using components supplied by Sharif Urethane Polymer Company, Iran, mixed in a carefully controlled ratio to optimize the polymerization and curing processes. This polymer binder served as the primary chemical stabilizer to enhance the engineering properties of the treated soils.

2.1.3 Waste tire textile fiber (WTTF)

WTTFs used in this study were obtained from a tire manufacturing facility in Ardabil, Iran. These fibers, originally used as reinforcement in tire cross-ply, were recovered by removing steel wires and shredding the tires, followed by separation from other residual materials. The recycled fibers were characterized by their orange color, odorlessness, and presence of residual recycled particles. Prior to testing, the WTTFs were processed to standardize their length and diameter. Fibers with diameters of 0.8 mm and lengths of 1, 2, and 3 cm were evaluated, exhibiting tensile strengths of approximately 600 MPa. Consistent with previous research (Pouramin et al., 2025), fibers measuring 2 cm were identified as optimal for reinforcing the PU-treated expansive clay, offering an effective balance between fiber-soil interlocking and minimizing fiber entanglement. Consequently, 2 cm WTTFs were selected to reinforce the PU-clay composite, resulting in improved strength and durability compared to other fiber lengths.

2.2 Sample preparation

In this study, bentonite soil was oven-dried for 24 hours before sample preparation. For PU-stabilized specimens, the optimum moisture content (OMC), determined through standard compaction tests, was divided into two portions. One portion

equaled half the weight of the PU and was used for its initial dilution, while the remaining portion accounted for the extra water needed to reach the target OMC. The polyurethane binder was prepared by mixing polyol and isocyanate components at a fixed mass ratio of 1:1.1. After blending the components for 30 seconds, the first portion of water (equal to half the PU weight) was added, followed by another 30 seconds of mixing. The resulting PU foam was allowed to expand for 5 minutes, during which time the second portion of water was thoroughly mixed into the dry soil. Subsequently, the PU foam and moistened soil were combined until a uniform mixture was achieved. This mixture was poured into cylindrical molds (38.3 mm diameter and 76.6 mm height) and statically compacted to reach maximum dry density. The mold dimensions were chosen in accordance with ASTM standards to maintain a 2:1 height-to-diameter ratio (Hatefi et al., 2024; Lal Mohammadi et al., 2023; Salimi et al., 2024b). Specimens were cured at room temperature for periods of 3 hours, 1 day, and 3 days. Based on the results, extending curing beyond 3 days did not significantly improve UCS. The optimal PU content was determined via mechanical testing. For fiber-reinforced samples, 2 cm long WTTFs were added to dry soil at dosages of 0.5%, 1%, 1.5%, 2%, 3%, and 4% prior to water and PU addition. After thorough mixing, the samples were molded and cured following the same procedure. After curing, selected specimens containing 8% PU and varying WTTF contents were sealed in plastic bags and stored at room temperature for 3 days before being subjected to a series of closed-system F–T cycles without moisture exchange (Aldaood et al., 2016). Each cycle consisted of 12 hours freezing at -16°C and 12 hours thawing at 18°C , replicating typical winter conditions in Iran (Ke et al., 2023; Santoyo and Baser, 2022). Samples underwent 1, 4, and 8 cycles based on prior studies showing that most degradation occurs during initial cycles, with minimal changes beyond eight (Ghazavi and Roustaie, 2010; Salimi et al., 2024b; Vakili et al., 2022). Following each F–T cycle, specimens were weighed and measured, then tested for UCS, and examined through microstructural analysis to assess durability and reinforcement effects.

2.3 Testing method

To evaluate the influence of F–T cycles on the performance of expansive clay treated with 8% PU and varying contents of WTTFs, a series of UCS and SEM tests were conducted. Each mixture was tested in duplicate to ensure repeatability and consistency, and the average values were reported along with error bars to reflect variability. UCS tests were performed in accordance with ASTM D2166 (2016), with samples loaded vertically at a constant deformation rate until failure. After mechanical testing, SEM analysis was conducted on untreated clay samples and those treated with 8% PU and 8% PU + 2% WTTF. Specimens were analyzed after 3 days of curing and after undergoing 8 F–T cycles. Prior to SEM imaging, the samples were fractured to expose fresh surfaces, coated with gold, and examined using a MIRA3 TESCAN microscope.

3 RESULTS & DISCUSSION

3.1 Unconfined compression tests

3.1.1 UCS

Initial strength tests revealed that incorporating PU into expansive clay significantly improved UCS, particularly after 3 days of curing. The UCS increased progressively with PU content, reaching a peak at 8%. However, further addition of PU beyond this threshold resulted in a decline in strength, likely due to polymer oversaturation and diminished bonding

efficiency between the polymer and soil particles (Zamanian et al., 2023; Zhang et al., 2019). Therefore, 8% PU cured for 3 days was selected as the optimal formulation for further durability testing, based on its superior short-term strength performance and material efficiency. As illustrated in Fig. 2, the UCS of untreated expansive soil decreased sharply with increasing F–T cycles, reflecting a cumulative loss of structural integrity due to crack formation and internal damage induced by ice crystal growth and meltwater pressure (Wang et al., 2021). The strength dropped by over 57% after 8 cycles, emphasizing the vulnerability of untreated soil to repeated climatic stress. In contrast, soil treated with 8% PU retained considerably more of its strength across F–T cycles. Although the UCS declined by about 30% after 8 cycles, the presence of the PU matrix significantly mitigated structural deterioration by enhancing internal cohesion and restricting the propagation of microcracks. This performance highlights the crucial role of PU in improving the F–T resistance of stabilized expansive clay. Further improvement was achieved by incorporating WTTFs. Samples reinforced with 2% WTTF exhibited the highest durability, maintaining greater UCS values than those with lower or higher fiber contents. The reinforcing fibers act as physical bridges within the soil–PU matrix, inhibiting crack expansion and facilitating energy dissipation under F–T stresses (Cheng et al., 2020; Li et al., 2018). However, increasing the fiber content beyond the optimal value led to performance loss, attributed to fiber clustering, increased internal voids, and disruption of the matrix homogeneity (Zamanian et al., 2023). The error bars in Fig. 2 represent the standard deviation derived from two repeated UCS measurements per mix design, ensuring that the reported trends are both reliable and reproducible despite limited replication. These findings confirm that a combined formulation of 8% PU and 2% WTTF provides a highly effective stabilization strategy, ensuring strength retention and durability of expansive clay under repeated F–T cycles.

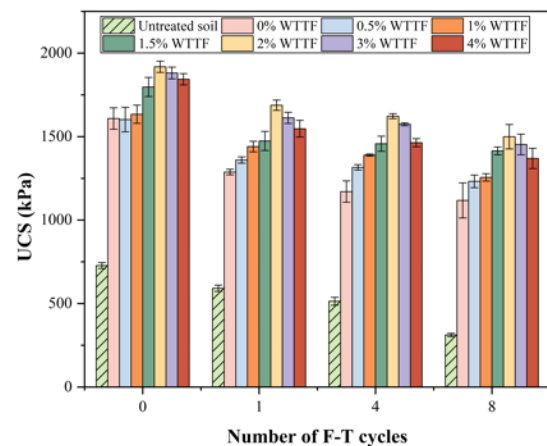


Figure 2. UCS of soil samples treated with 8% PU and different WTTF contents under F–T cycles.

3.1.2 Mechanical moduli under F–T cycles

The effects of F–T cycles on the mechanical behavior of stabilized expansive clay were evaluated through two key parameters, namely the energy absorption capacity (E_u) and the secant modulus (E_{50}), as shown in Figs. 3a and 3b.

E_u , derived from the area under the stress–strain curve up to peak stress, reflects the material's energy dissipation capacity during deformation. E_{50} , calculated at 50% of peak stress, represents overall stiffness (Salimi et al., 2024b). Untreated soil

exhibited low values for both moduli due to its inherently ductile nature and weak inter-particle bonding, with E_u and E_{50} at approximately 20 kJ/m³ and 298 MPa, respectively. Incorporating 8% PU significantly enhanced both parameters to 22 kJ/m³ and 416 MPa, indicating improved cohesion and stiffness. The addition of 2% WTTFs to the 8% PU-treated soil led to a notable enhancement in both moduli, with E_u increasing to 32 kJ/m³ (a 60% rise) and E_{50} reaching 624 MPa (a 109% improvement), underscoring the complementary effect of fiber reinforcement alongside PU stabilization. The reinforcing role of WTTFs becomes evident through increased stiffness and energy absorption. Fibers act as micro-bridges, restricting crack propagation and improving load transfer (Roustaei et al., 2024; Salimi et al., 2024b). However, fiber contents exceeding 2% caused a decline in E_u and E_{50} , likely due to fiber agglomeration and excessive ductility that weakened the composite (Tran et al., 2018). Exposure to F–T cycles led to a general reduction in E_u for all specimens. The untreated soil showed a 35% decrease, while PU-treated samples exhibited more moderate losses. As an illustration, E_u declined by 27% in the sample stabilized with 8% PU (from 22 to 16 kJ/m³), while a 25% reduction was observed in the 8% PU + 2% WTF specimen (from 32 to 24 kJ/m³), suggesting that although fiber inclusion contributes to damage control, it does not entirely eliminate the adverse effects of F–T cycles. Interestingly, E_{50} values for PU-treated samples increased slightly after F–T exposure. The 8% PU specimen showed a 10% increase (from 416 to 457 MPa), while the 8% PU + 2% WTF sample rose from 624 to 677 MPa (8.5% increase), likely due to reduced strain at failure and enhanced interfacial bonding (Salimi et al., 2024b). This suggests that PU and WTTFs contribute not only to initial stiffness but also to improved resilience under cyclic thermal stress.

3.2 SEM-based microstructural analysis

The morphological characteristics of untreated and stabilized expansive clay samples following exposure to 8 F–T cycles are illustrated in Figs. 4a–c. These SEM images provide detailed insights into how different treatments influence the soil's microstructure under cyclic freezing and thawing conditions. As illustrated in Fig. 4a, the untreated sample exhibits a severely degraded microstructure, with prominent voids and wide cracks highlighting extensive internal damage. The disruption is primarily attributed to repeated ice formation and melting, which cause expansion and contraction of pore water. This process loosens the particle bonds, increases porosity, and ultimately weakens the mechanical strength of the soil. By contrast, Fig. 4b illustrates that the sample treated with 8% PU develops a more cohesive and structurally stable matrix. While some microcracks are observed, the PU forms a continuous binding matrix that holds the particles together and prevents major structural breakdown. The polymer coating reduces the formation of large pores and effectively limits moisture-induced expansion, which helps retain the soil's integrity after multiple F–T cycles. Fig. 4c demonstrates that the specimen treated with 8% PU and 2% WTTFs exhibits the most resilient and well-integrated structure. Although some increase in porosity is evident compared to pre-F–T conditions, the presence of WTTFs reinforces the soil matrix by forming bridges between particles and restricting crack propagation. The fibrous texture, with its rough and scaly surface, enhances interfacial bonding and mechanical interlock. This synergy between PU and WTTFs ensures that the fibers remain embedded within the matrix even after cyclic temperature changes, reducing the likelihood of fiber pullout and maintaining structural cohesion. A comparison with the findings of Roustaei et al. (2015) highlights the improved

performance of the current stabilization approach. In their study, F–T cycles led to noticeable detachment between fibers and soil particles in untreated reinforced specimens, resulting in reduced cohesion. In contrast, the use of PU in this research appears to mitigate such separation by firmly binding the WTTFs to the surrounding soil matrix, thereby preserving the internal cohesion and structural stability of the composite even after repeated F–T exposure.

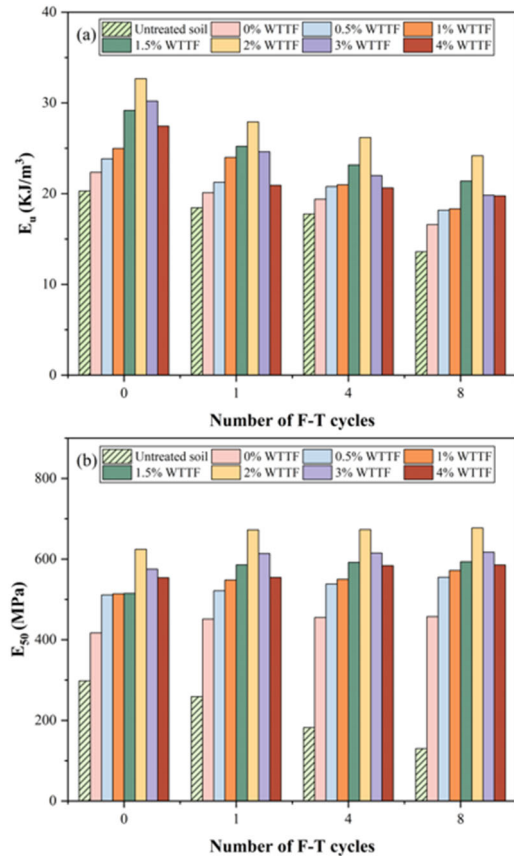


Figure 3. Effects of different WTTF contents on (a) E_u and (b) E_{50} of untreated and 8% PU-stabilized expansive clay after 0, 1, 4, and 8 F–T cycles.

4 CONCLUSIONS

This study investigated the effects of F–T cycles on the mechanical performance and durability of expansive clay stabilized with PU and reinforced with varying WTTF contents. UCS and SEM tests were conducted to comprehensively assess the performance of treated soils exposed to F–T cycles. The key findings are summarized below:

- The combined application of PU and WTTFs substantially improved the UCS of expansive clay, particularly under F–T conditions. The optimum formulation was identified as 8% PU and 2% WTTF, which yielded the highest compressive strength and overall structural integrity. This synergy between the chemical stabilization provided by PU and the mechanical reinforcement from WTTFs proved highly effective in mitigating strength loss during F–T cycling.
- The inclusion of PU and WTTFs also enhanced the soil's E_u and E_{50} , contributing to greater toughness and stiffness. Although repeated F–T cycles led to a general decline in E_u and E_{50} across all samples, those treated with PU and reinforced with WTTFs exhibited significantly better resilience. This was primarily due to the bridging

mechanism of fibers, which limited the growth of microcracks and distributed stress more uniformly.

- SEM images after 8 F–T cycles showed severe microstructural damage in untreated clay. PU treatment reduced cracks and maintained cohesion, while adding 2% WTTF further reinforced the structure by bridging particles and preventing separation. This confirms the effectiveness of PU–WTTF stabilization under F–T conditions.

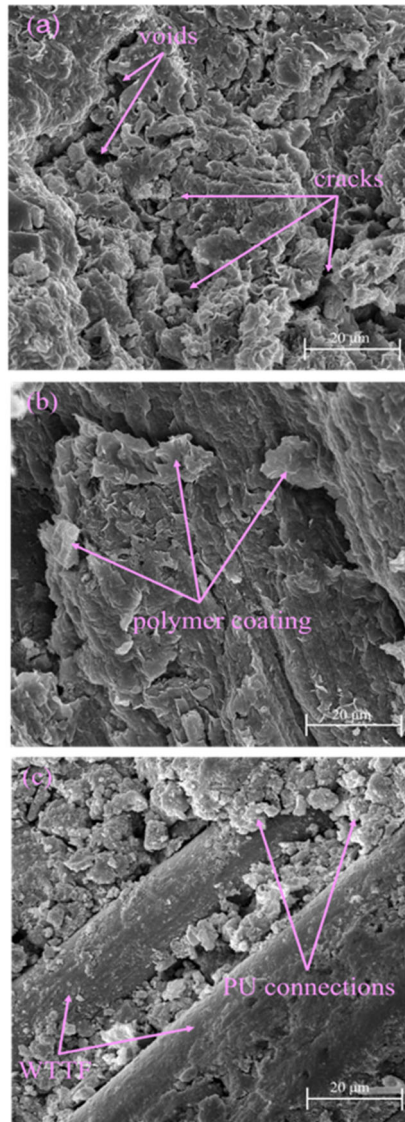


Figure 4. SEM images after 8 F–T cycles: (a) untreated soil, (b) 8% PU-treated soil, and (c) 8% PU + 2% WTTF-treated soil.

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