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Shear Strength Durability Investigation for Gypseous Soil Enhanced by Pectin Biopolymer

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Biopolymers; Durability; Gypseous soil; Pectin; Soil Improvement; Wetting and Drying.

Highlights:

- Gypseous soil improvement using biopolymers as additives.
- Shear strength of soil improved by pectin biopolymer.
- Durability against wetting and drying cycles of improved gypseous soils.

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Abstract: Several studies have shown that biopolymers improve soil; however, they mostly looked at how the shear strength and collapsibility improved without looking at how long the treatments would last. However, durability is an important factor that should be considered to get the most out of the treatment. This study investigates the durability and strength of gypseous soil enhanced by pectin biopolymer with periodic changes in wetting and drying. Soil with a gypsum content of 40% was mixed with 2% pectin biopolymer, and the samples were passed through successive wetting and drying cycles (1, 5, 10, and 15). The results indicated that periodic wetting and drying of pectin biopolymer-treated gypseous soil increased the shear strength of the soil until cycle 5, after which there was a slight and gradual decrease in strength until cycle 15 due to dissociation of pectin monomers under hydration and incomplete re-formation during re-drying, with a force decreasing by about 22% till ten cycles. Even beyond several cycles, some degree of strength and strength restoration could be observed. Also, the volumetric stability of the improved samples was clear until the last wetting and drying cycle.

دراسة ديمومة مقاومة القص للتربة الجبسية المحسنة بالبكتين الحيوي

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الخلاصة

أظهرت العديد من الدراسات أن البوليمرات الحيوية يمكن أن تساعد في تحسين التربة، لكنها بحثت في الغالب في كيفية تحسن مقاومة القص وقابلية الانهيار دون النظر إلى (ديمومة) المدة التي ستستمر فيها المعالجات. ومع ذلك، تعد الديمومة عاملاً مهماً يجب أخذه بعين الاعتبار للحصول على أقصى استفادة من المعالجة. في هذه الدراسة تم تقييم مقاومة وديمومة التربة الجبسية المعالجة بالبوليمر الحيوي البكتين تحت ظروف الترطيب والتجفيف الدوري. تم خلط التربة التي تحتوي على الجبس بنسبة ٤٠٪ مع ٢٪ من البوليمر الحيوي البكتين، وتعرض العينات إلى دورات متتالية من الترطيب والتجفيف (١، ٥، ١٠، ١٥). تشير النتائج التي تم الحصول عليها إلى أن الترطيب والتجفيف الدوري للتربة الجبسية المعالجة بالبوليمر الحيوي البكتين يؤدي إلى زيادة مقاومة القص للتربة حتى الدورة الخامسة وبعدها يحدث انخفاض طفيف وتدرجي في المقاومة حتى الدورة ١٥ وذلك بسبب تفكك مونومرات البكتين، تحت الترطيب وإعادة التكوين غير الكاملة أثناء إعادة التجفيف، مع انخفاض في المقاومة بنسبة ٢٢٪ تقريباً خلال ١٠ دورات. ومع ذلك، لوحظ وجود درجة معينة من المقاومة واستعادة المقاومة حتى بعد عدة دورات. كما أن الثبات الحجمي للعينات المحسنة كان واضحاً حتى دورة الترطيب والتجفيف الأخيرة.

الكلمات الدالة: البوليمرات الحيوية، متانة، التربة الجبسية، البكتين، تحسين التربة، الترطيب والتجفيف.

1. INTRODUCTION

In recent years, geotechnical engineers have concentrated on developing biological treatments to enhance soil behavior and ground improvement methods to boost sustainability engineering and reduce environmental troubles related to high carbon dioxide emissions from cement manufacturing in geotechnical engineering applications [1]. One of the recent approaches and sustainable solutions for soil enhancement and ground modifications is improving biopolymer additions. Biopolymer treatment for soils has achieved substantial enhancement and successful field-scale implementation in geotechnical properties in many types of soils, such as increasing shear strength and reducing permeability and erosion [2]. Gypseous soil is one of the problematic soils. Its problem is dissolving gypsum matter (calcium sulfate) between the soil particles, losing their bonds when immersed in water. This process creates large voids, leading to serious deformations and collapse in the gypseous soil fabric [3]. Theoretically, petroleum-based and bio-based polymers are biodegradable. The rate of degradation varies according to the polymer composition. A slow degradation rate for polymers can be considered durable or non-biodegradable [4]. Therefore, the study of the long-term behavior and durability of soils treated with biopolymers against biodegradation, basically under wetting and drying cycles, must be confirmed to evaluate the usage of biopolymers in geotechnical engineering applications [5-6]. Theyab et al. [7] evaluated the durability and strength of the gypseous soil enhanced by adding xanthan biopolymer. Three gypseous soil types with various gypsum contents were selected and tested. Three percentages of xanthan gum biopolymer 2, 4, and 6% were added to these gypseous soils to modify them. Their results showed that the maximum dry density

decreased by adding xanthan gum, whereas an increase in the optimum moisture content was noted. A decrease in the soil collapse potential by about 30% to 45% was observed by adding xanthan gum. A significant gain in shear strength was detected by the direct shear results for the gypseous soil-treated xanthan gum biopolymer. The effect of casein biopolymer on gypseous soil was studied by Theyab et al. [8]. Three primary gypseous soil characteristics were studied, i.e., compaction properties, shear strength, and collapse potential. Gypseous soils were improved by adding different percentages of casein biopolymers. Their results indicated that casein biopolymer decreased the maximum dry unit weight while increasing the optimum water content. Collapse potential was reduced between 65 and 80% compared to the untreated soil. Gypseous soil enhanced by casein biopolymer showed an increase in shear strength in soaked and dry test conditions. Mutar [9] studied the gypseous soil durability enhanced by xanthan gum or casein biopolymers. The study examined the durability of improved gypseous soil by adding 6% casein or 2% xanthan after several cycles of soaking and drying and periodic cycles of freezing and thawing. The author detected a decrease in the shear strength of improved soil with increasing the number of cycles for the two conditions, soaking-drying and freezing-thawing for both biopolymers. Also, a decrease was noted in the volume change after cycle 12. Compared to the two biopolymers in terms of durability, xanthan gum was more effective and stable than casein biopolymer. Hussein et al. [10] studied improving gypseous soil by adding pectin biopolymer in different concentrations (0.5%, 1%, and 2%) to four soil samples that have a gypsum content of 10%, 20%, 40%, and 62%. The chemical and mechanical properties of the soil-pectin mixture were investigated.

The results revealed a significant reduction in Calcium Hardness (CH) and collapse potential (Cp) values attributed to bio-gel encapsulation of soil particles and void-filling properties. The reduction percentages in the calcium hardness values reached 0.67, 73, 75, and 68% for soils 1, 2, 3, and 4, respectively. Collapse potential values decreased by 0.63, 0.63, 0.65, and 0.7 for the four soils. In another study conducted by Chang et al. [11], the durability of the strength of Korean sand was improved using Gellan gum biopolymer as an additive against cycles of soaking and drying. The study results revealed that the cyclic soaking and drying of the sand treated with Gellan gum gradually degraded strength. The degradation in strength may be due to the dismantling of the Gellan gum monomers under soaking and incomplete re-composition through the re-drying phase, with a reduction in strength reaching about 30% over ten cycles. Lee et al. [12] studied the effect of cyclic soaking-drying and freezing-thawing on the shear strength durability of soils modified by adding biopolymers. A gradual deterioration in the soil shear strength was noticed with the advance of soaking and drying cycles as well as freezing-thawing cycles due to water adsorption and dilution of biopolymers locally. Sand with poorly graded particles was exposed to these weathering effects; however, this problem may be attenuated when the soil has a fine content of 15–25%. Meanwhile, the durability of gypseous soil treated with pectin biopolymer is yet to be studied. Thus, the major objective of this article is to examine the durability of biopolymer-enhanced gypsum soils with cyclic wetting and drying processes. A gypseous soil with 40% gypsum content was selected and improved by two percent replacement of soil weight with pectin biopolymer. Moreover, the normal and biopolymer-modified samples were tested for shear strength and unconfined compressive strength tests. Since pectin has emerged as a biopolymer with a substantial ability for soil stabilization, studying its durability is

important when using it in geotechnical engineering applications.

2. MATERIALS AND METHODOLOGY

2.1. Gypseous Soil

A Soil specimen was extracted by digging 1 m from ground level. The site where the specimen was collected from is located in Tikrit University campus. Gypsum content for the specimen was chosen to be 40 %. This range represents the average gypsum content for the construction depth at that site, ranging between 0.5 m to 1.5 m depth from the ground surface. Table 1 and Table 2 illustrate the chemical and physical characteristics of the soil specimen, respectively. Sieve analysis was conducted on the soil specimen, as shown in Fig. 1. According to the sieve analysis results, the soil fell into group SP, poorly graded sand, based on the Unified Soil Classification system (ASTM D2487 – 17).

Table 1 Chemical Properties of the Soil Specimen.

Properties	Value
Gypsum content %	40
Total soluble salts (T.S.S)%	52
Organic matters (O.M)%	0.21
pH	7.6

Table 2 Parameters of Physical Characteristics for the Soil.

Properties	ASTM Standard	Value
Unified Soil Classification system		SP
Gravel %		3.3
Sand %		93.3
Fines %	ASTM D2487-17	3.4
Coefficient of uniformity (Cu)		5.6
Coefficient of curvature (Cc)		0.65
Specific gravity (Gs)	ASTM D854	2.54
Atterberg's limits (LL)%	ASTM D4318	27
(PL)%		N.P
Unit weight in field (γ_{field}) kN/m ³	ASTM D1556	13.4
Maximum dry unit weight ($\gamma_{dry,max}$) kN/m ³	ASTM D1557	16.4
Minimum dry unit weight ($\gamma_{dry,min}$) kN/m ³	ASTM D1557	11.13
Optimum moisture content (O.M.C)%	ASTM D1557	11.5

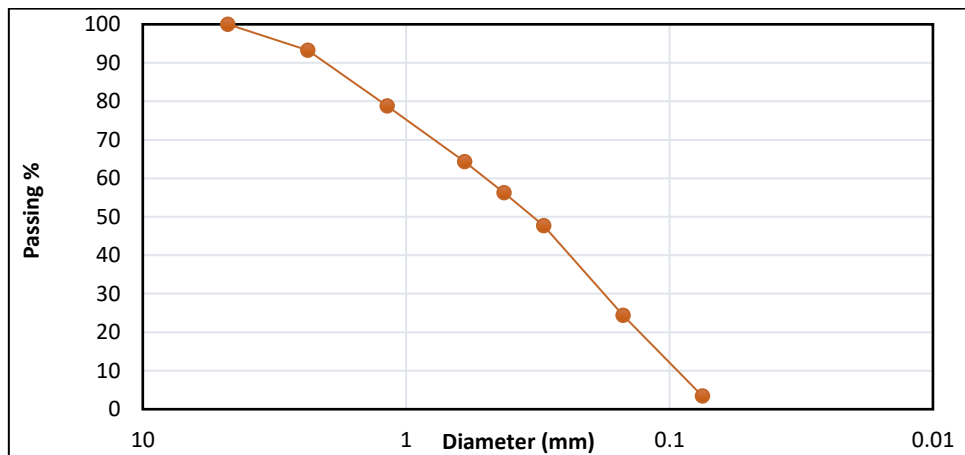


Fig. 1 Particle Size Distribution Curve for Soil Specimen.

2.2. Pectin Biopolymer

The pectin family of biopolymers is mostly made up of negatively charged polysaccharides held together by 1,4-linked D-galacturonic acids. Many applications of these compounds can be found in food, pharmaceuticals, personal care and polymers as emulsifiers, gelling agents, glazing agents, stabilizers, and/or thickeners. There are two types of commercial pectin: high methylated pectin with a methylation level greater than 50% and low methylated pectin with a methylation level lower than 50% [13]. By aminolyzing high methoxy pectin, low methoxy pectin can be obtained. Hydrophobic forces between methyl groups and hydrogen bonds cross-link methyl pectin in high co-solute concentrations and low pH conditions to form gels with high methoxy pectin levels. Unlike Low Methoxy Pectin, Low Methoxy Pectin forms ionic bonds through calcium bridges between carboxyl groups from adjacent chains. During low shear rates, pectin gels display Newtonian behavior, whereas shear-thinning occurs when the shear rate increases. Several articles, such as [14], provide an overview and more details about pectin's physicochemical properties. This study was performed with commercial pectin powder, as illustrated in Fig. 2.

3. PREPARATION OF SOIL-TREATED SAMPLE

According to a previous study by Hussein [15], the typical value of the pectin additive was 2% of the sample weight for the gypsum soil of 40% gypsum content. A pectin solution was prepared by taking a 2% pectin of the soil specimen's weight and mixing it with water equal to the volume of optimum moisture content for the same specimen properly in the magnetic mixing device. Pectin solution mixed with soil specimens to compound a soil-pectin mixture. The mixture of enhanced soil by pectin was remolded in three layers in the direct shear mold with dimensions of (50×50×20 mm). Also, it was remolded in five layers to form samples for an unconfined compression test in a mold with dimensions of 100 mm height and

43 mm diameter, as illustrated in Fig. 3. All remolded samples were prepared by adopting the optimum moisture content and maximum soil dry unit weight. The period for treatment of the samples before testing was investigated to find the period in days that gives the typical applicable time to reach the maximum percent of curing for the soil-pectin mixture samples. Samples were cured for different periods (7, 14, and 28 days), and the shear strength parameters for each period were calculated. The relationship between cohesion values and curing time is shown in Fig. 4, whereas the relation between the angle of internal friction and curing time is illustrated in Fig. 5. According to these results, the practical period for curing was 14 days for all samples. After the treatment, the samples were extracted from molds and passed through several cycles of soaking and drying. The number of cycles adopted at each test were 1, 5, 10, and 15. All the improved specimens sustained several successive soaking and drying cycles. Laboratory tests, i.e., direct shear and unconfined compressive strength, were performed on improved and unimproved specimens after each number of adopted cycles. These tests were conducted to investigate the variation of soil's shear strength and detect its durability.



Fig. 2 Commercially Produced Pectin Powder Used in this Study (Purchased from the Laboratory Materials Offices in Bab Al-Moadham in Baghdad, Iraq).



Fig. 3 Remolded Samples for Unconfined and Direct Shear Tests.

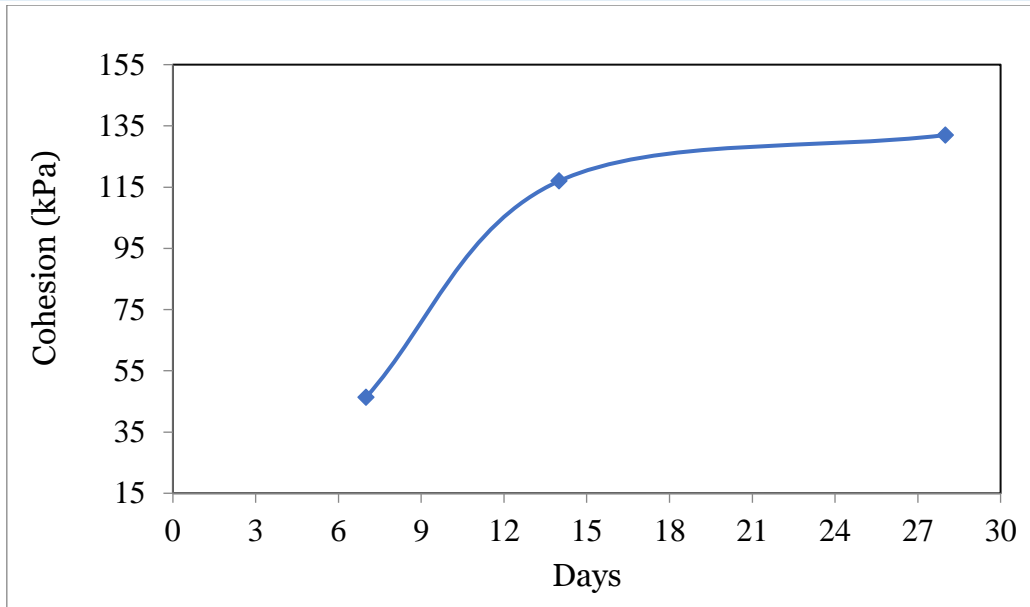


Fig. 4 Curing Time and Cohesion Relationship for Remolded Specimens.

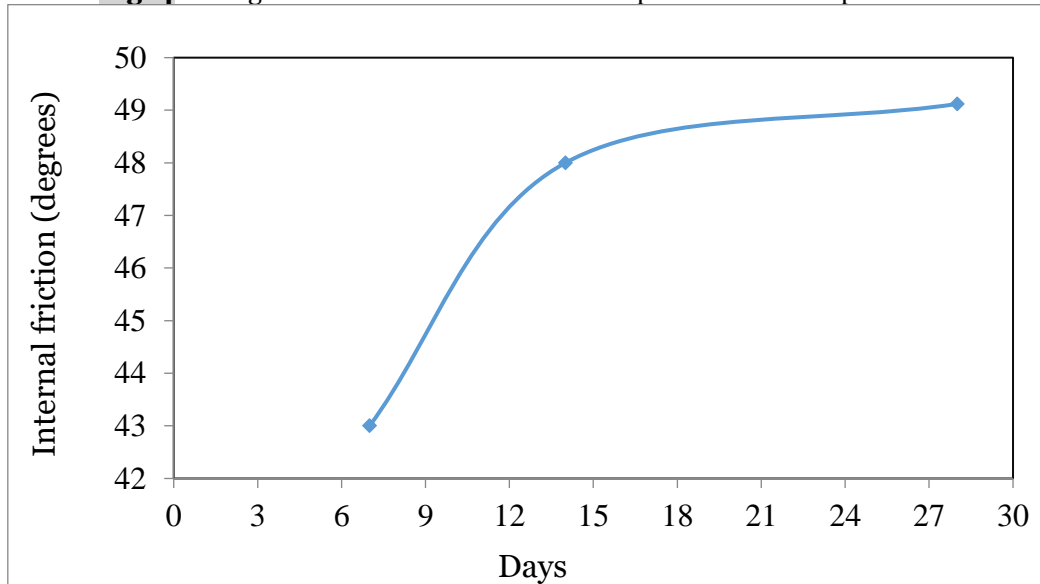


Fig. 5 Curing Time and Internal Friction Angle Relationship for Remolded Specimens.

4. RESULTS

4.1. Shear Strength Durability of Pectin-Treated Samples

Figure 6 shows the shear strength relationship with the progression of soaking-drying cycles at a vertical stress of 50 kPa. The curve shows the increase in shear strength after the first cycle. The increase continued by a large percentage compared to the strength at the zero cycle, where the percentage increase was 152% at the fifth cycle since the shear strength changed from 232.6 to 665 kPa. This large increase may be due to water absorption and forming hydrogels, resulting in additional hydrogel hydration, pectin biofilms condensation after wetting and drying, pore filling, and interference between the treated soil particles and forming the sand-pectin matrix with a single mass and a large contact area, increasing the stress cycles. The shear is required to break

the contact and overlap in the sand-pectin matrix, increasing the shear strength.

After the wetting and drying cycles, the shear strength decreased after the fifth cycle and continued to decrease until the 15th cycle by 52%; however, the shear strength remained higher than at the zero cycle, where the shear strength at the 15th cycle was 304 kPa, while it was 263.2 kPa at the zero cycle. This decrease was that the outer monomers of pectin bio-gel absorb water molecules, separate after increasing soaking cycles, and separate from the base gel body. On the other hand, the inner monomers maintained their strength and structure to some extent. The large number of soaking and drying cycles separated a larger amount of monomers from the gel body of the pectin and, consequently, a continued decrease in the shear strength.

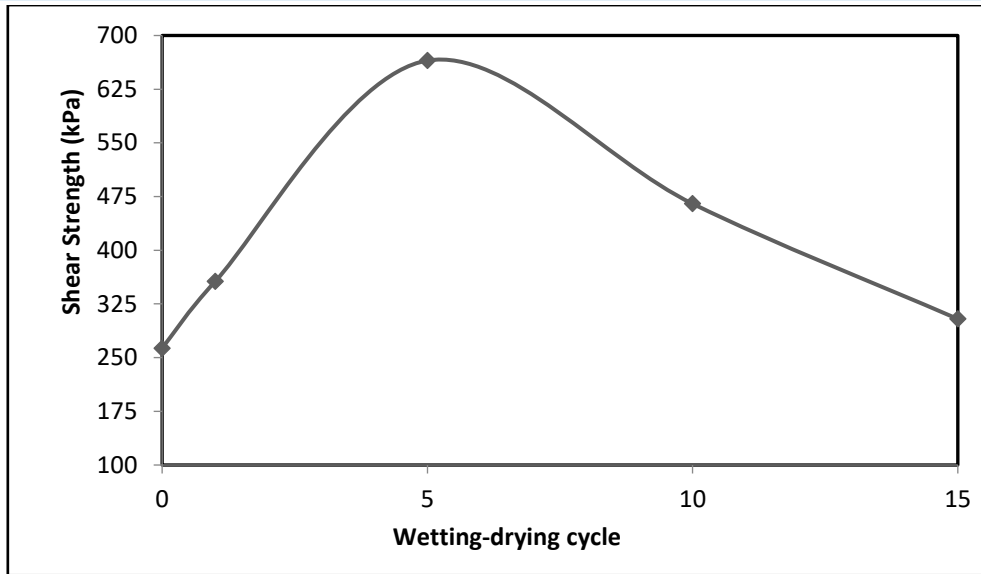


Fig. 6 Relationship between Shear Strength and Wetting -Drying Cycles at a Vertical Stress of 50 kPa for Treated Soil Samples.

Figure 7 illustrates the shear stress relationship with the normal stress of the soil specimens treated with pectin after the progression of wetting and drying cycles. The figure shows the cohesion and the friction angle increase until the fifth cycle compared to the zero cycle. After the fifth cycle, the grip decreased with a slight increase in the friction angle. Table 3 shows the internal friction angle and cohesion with the succession of wetting and drying cycles. The strength of the previously mentioned sand-pectin matrix continued until the fifth cycle and increased the cohesion and the angle of internal friction until the fifth cycle; this behavior is clear in Fig. 8, which shows the shear stress relationship with the horizontal displacement (H-Disp.). The fastest peak at the fifth cycle of 679 kPa at 3 mm horizontal displacement. After the fifth cycle, the strength became 480 kPa at

the tenth cycle and decreased to 300 kPa at the fifteenth cycle at 3.5 mm and 2.5 mm horizontal displacement, respectively. Then, the external monomer separation of the pectin gel decreased in cohesion and friction angle. The decrease in cohesion continued with the immersion and drying cycles and the decrease in the bio-gel thickness.

Table 3 Shear Parameters of Treated Samples with 2% Pectin with Soaking and Drying Cycles Progression.

No. of Cycles	ϕ (degrees)	c (kPa)
0	41	152.13
1	40	268.92
5	47	488
10	39	369.33
15	37.4	126

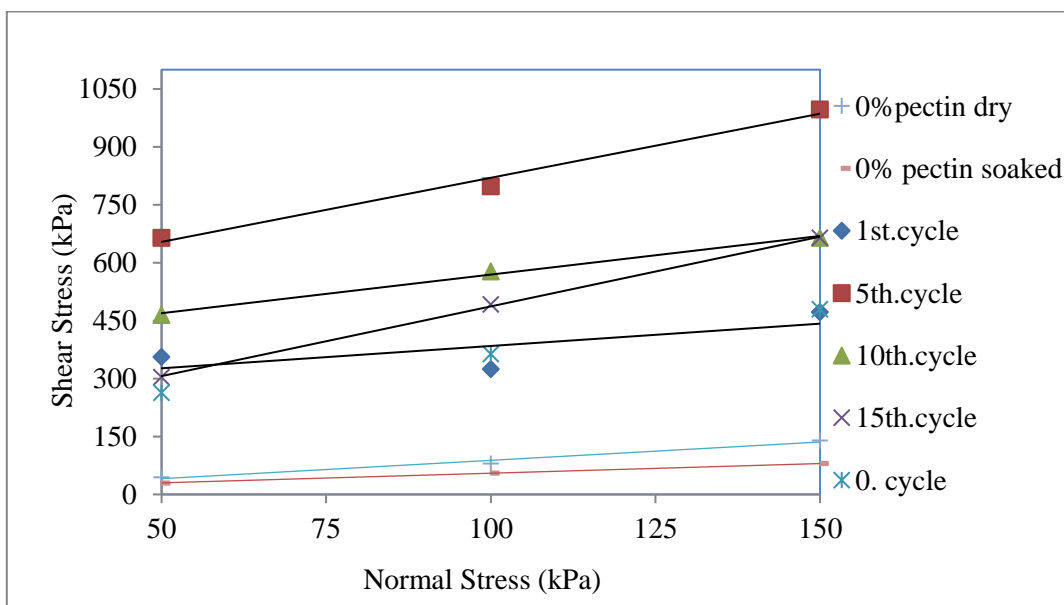


Fig. 7 Shear Stress Relationship Normal Stress for Pectin Treated Soil Sample with the Progression of Wetting and Drying Cycles.

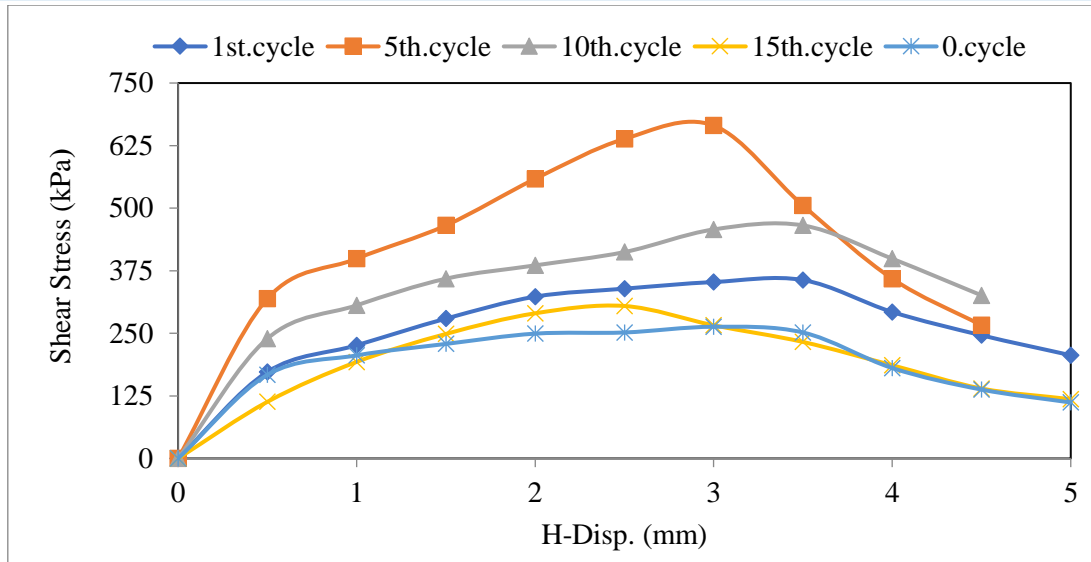


Fig. 8 Horizontal-Displacement and Shear Stress Relationship for Wetting and Drying Cycles of Pectin Treated Soil.

4.2.UCS Durability of Pectin-Treated Samples

The curve in Fig. 9 shows the unconfined compressive stress (UCS) with the wetting and drying cycles of soil samples treated with pectin to study their durability. The curve shows the increase of UCS up to the fifth cycle, where the UCS was 948 and 1210 kPa for the first and fifth cycles, respectively, while it was 914 kPa before the soaking and drying cycles. The rise in resistance happened because the remolding of samples with the optimum moisture content was insufficient to create a bonding force between soil particles. After being soaked in water, the pectin hydrogels absorbed the water molecules and expanded to fill the pores more by encapsulating the soil grains. Upon drying, the viscosity of the bio-gel increased, resulting in greater cohesion and condensation of the pectin bio-membranes that work with the soil particles as a single mass, thus increasing the UCS to failure. After the fifth cycle, the UCS decreased and continued until the 15th cycle of the soaking and drying cycles, when it

decreased to 822 and 705 kPa for the 10th and 15th cycles, respectively. By increasing the soaking and drying cycles, the monomers of the bio-gel were subjected to separation from the main gel body, thus reducing the main gel thickness and reducing the maximum UCS to failure. The stress-strain relationship curve in Fig. 10 for UCS samples treated with pectin after 15 wetting and drying cycles. For pectin to allow more bio-gel to participate in the binding of soil particles and with drying, the condensation and agglomeration of the biofilm increased besides its stiffness, thus increasing the cohesion of the samples, resulting in a higher E_{50} that reached 70.5 MPa at the fifth cycle. After the fifth cycle, the elastic stiffness decreased to 16.4 and 15.7 MPa at the 10th and 15th cycles, respectively, due to the main gel thickness decrease after separating part of the outer gel monomers. Continuing the soaking cycles and upon drying, the biopolymer films shrank and become more brittle, reducing the elastic stiffness.

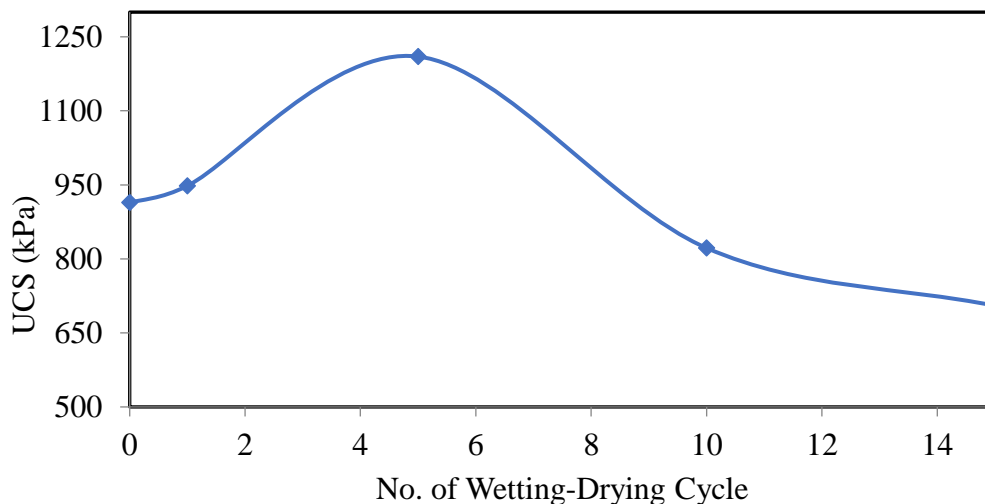


Fig. 9 Unconfined Compressive Strength with the Wetting and Drying Cycles of Pectin Treated Soil.

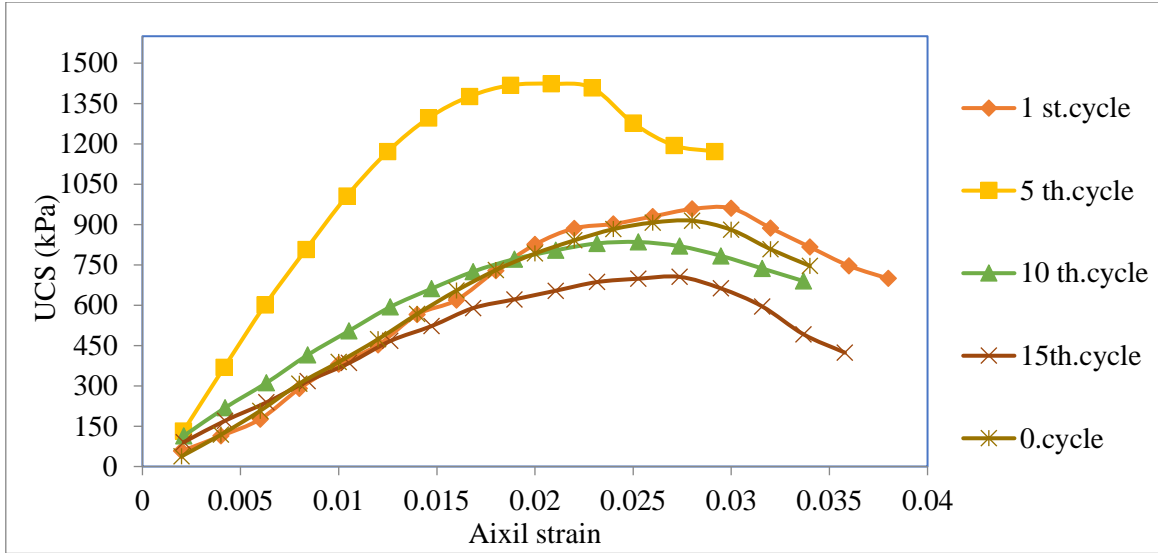


Fig. 10 Stress Relationship with Strain for the Wetting and Drying Cycles of Pectin Treated Soil.

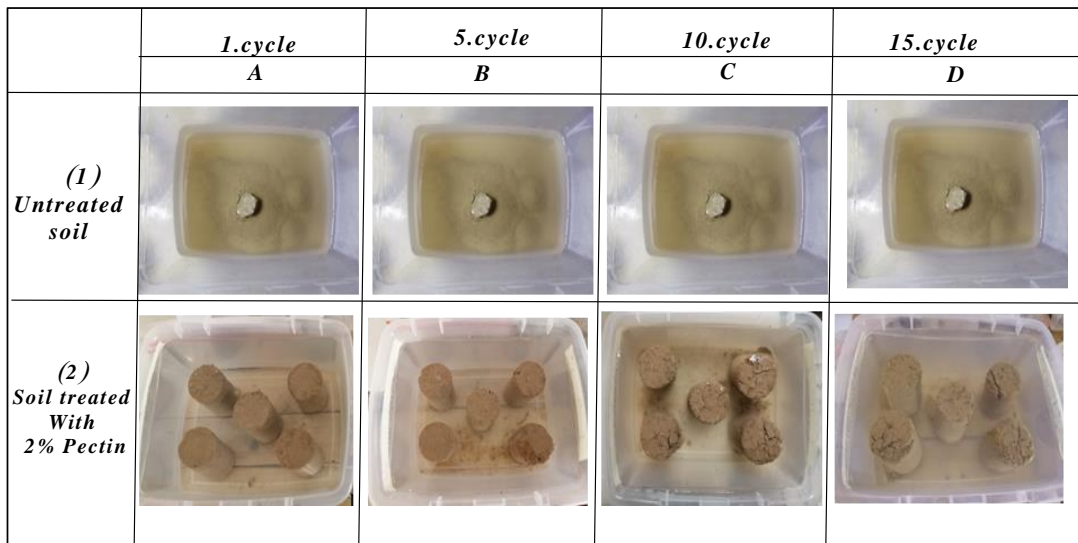


Fig. 11 Sample Shapes with Advancing in the Soaking and Drying Cycles for Enhanced and Natural Soil Samples.

4.3. Wetting-Drying Cycles for Untreated and Treated Soil

The stages of wetting and drying of untreated soil samples and treatment with Pectin are illustrated in Fig. 11. It can be observed, through the shapes A1-C1 in Fig. 11, a complete breakdown of the untreated soil samples, and the collapse was within 10 minutes of the beginning of the first wetting cycle. Once the samples were wetted in water, the connections between the soil particles began to dissolve very quickly, causing them to collapse completely. Plates A2 to C2, illustrated in Fig. 10, show the wetting-drying cycle effect on the shape and stability of the samples treated with pectin. It is clear that the samples maintained their shape and showed resistance and durability against deformation and deterioration with the proceeding of wetting and drying cycles; however, the untreated samples deformed directly from the first cycle of wetting. After cycle No. 10 of soaking and drying, the

enhanced specimens showed a simple crack on the top of the specimens.

5. CONCLUSIONS

The main conclusions of the present study could be summarized as follows:

- The durability of gypseous soil specimens that improved with pectin showed a development in their shear strength up to 150% until the fifth cycle of wetting and drying, and the rate of increase dropped after that to 15.5% at cycle number 15. When the specimen was dried, the strength was fully restored.
- The durability of the improved samples by pectin was more cohesive and stable due to the nature of the biological monomers that make up the polymeric chains of pectin.
- The unconfined compression strength increased up to 32.4% at the fifth cycle, where they were much higher than the values before the soaking and drying

cycles, and then began to decrease slightly and steadily up to 22.9% at the 15th cycle.

- Untreated gypseous soil samples are directly damaged when immersed in water, unlike the gypseous soil treated with pectin biopolymer.
- Restoring the formation of the biomonomers completely upon drying increased the hardness of the biohydrogel, which in turn increased the strengthening.
- Treatment of gypseous soil by pectin biopolymer can be adopted since it showed acceptable durability against wetting and drying cycles.

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NOMENCLATURE

C_c	Coefficient of curvature
CH	Calcium Hardness
C_p	Collapse potential
C_u	Coefficient of uniformity
G_s	Specific gravity
LL	Liquid Limit, %
OM	Organic matters, %
$OM.C$	Optimum moisture content, %
pH	Hydrogen number
PL	Plastic limit, %
$T.S.S$	Total soluble salts, %
UCS	Unconfined compressive strength
Greek symbols	
c	Soil cohesion, kPa
ϕ	angle of internal friction, degrees
$\gamma_{dry) max}$	Maximum dry unit weight, kN/m ³
$\gamma_{dry) min}$	Minimum dry unit weight, kN/m ³
γ_{field}	Field dry unit weight kN/m ³

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