Splitting tensile strength and microstructure of xanthan gum-treated loess

Junran Zhang (✉ zhangjunran@ncwu.edu.cn)

North China University of Water Conservancy and Electric Power

Tong Jiang

North China University of Water Conservancy and Electric Power

Jindi Zhao

North China University of Water Conservancy and Electric Power

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Abstract

The tensile strength of loess is closely related to geological disasters. As an eco-friendly material, biopolymers have an excellent strengthening effect on the mechanical properties of soil. The effect of different initial dry densities and Xanthan gum (XG) contents on the microstructure and mechanical behavior of XG-treated loess was studied through a series of microscopic tests and splitting tensile tests based on the PIV technique. The results show that during the dehydration process, the XG became concentrated and agglomerated, forming bridge links between soil particles and covering the surface of soil particles. The XG-treated loess had a major concentration of micropores and mesopores, with greater peak intensities of pore size distribution curve than untreated loess. The load-displacement relation curves of specimens with different XG contents and initial dry densities showed strain-softening. The displacement vector field indicated that the primary cracks of specimens were radial vertical, the secondary cracks were well developed. With the increase of the XG content and initial dry density, the strain-softening phenomenon was more significant, the splitting tensile strength and brittleness of specimens were greater. XG treatment made the soils obtain stronger cementation and denser structure, which helped to increase strength and brittleness.

Introduction

The loess has the characteristics of high silty content, low natural water content, large porosity, small natural tensile strength and easy collapsibility when exposed to water. It is easy to produce a differential settlement, cracking and collapse when directly used as engineering filler, which is challenging to meet the requirements of actual construction^{1,2}. As one of the three significant strengths of soil, tensile strength is often neglected because of the relatively weak tensile resistance of the soil. However, geological disasters and engineering accidents such as ground cracks, landslides, earth-rock dam cracking and slope instability are related to the low tensile strength of loess³⁻⁶. Therefore, it is of great practical significance to reinforce loess and study the modification effect on the tensile properties of loess.

Cement, lime and fly ash have been widely used in engineering construction in the loess areas. The geotechnical properties of soil can be improved by these modified materials either by single addition or combined incorporation⁷⁻⁸. Nevertheless, the production of these materials is accompanied by a large amount of dust and wastewater, which seriously pollutes the environment and harms human health. Under the call of national green environmental protection, a series of techniques to stabilize loess emerged at the historic moment. Root, wheat straw, lignin fiber, fiber yarn, glass fiber, polypropylene fiber and basalt fiber have been used as modified materials for soil stabilization⁹⁻¹⁴. The improvement of engineering properties of loess by stabilizing agents such as F1, HEC, SH and nano-SiO₂ has also been explored¹⁵⁻¹⁸. Liu *et al.*¹⁹ pointed out that microbial-induced carbonate precipitation (MICP) is widely used in civil engineering. However, these technologies are undesirable for various engineering practices because of their high cost, stringent environmental requirements and the complete dependence on the

material itself in terms of strength improvement²⁰. Therefore, it is necessary to explore an economic, eco-friendly and universal soil treatment technique.

Existing studies have shown that biopolymers have excellent performance in soil reinforcement, promoting vegetation growth and desert control²¹⁻²³. As one of the biopolymers, Xanthan gum (XG) can provide higher unconfined compressive strength than guar gum and β -glucan²⁴. Chang *et al.*²⁵ pointed out that adding a small amount of XG (1%) could enhance the erosion resistance of Korean red loess. Compared with wet mixture, dry mixing can get higher material strength. Fatehi *et al.*²⁶ pointed out that when XG content exceeds 2%, the strength of XG-treated soil tends to decline. Lee *et al.*^{27,28} modified sand with XG and found that the shear strength of the specimens was improved best under dehydrated XG conditions. Chang *et al.*²⁹ proposed that the interaction between XG and soil particles can be entirely played under low water content. Loess exists widely in a semiarid to arid climate with low natural water content, which provides suitable conditions for XG application in loess improvement. In addition, XG can increase the fatigue life of soil³⁰ and reduce the rheological property, permeability and dispersion of soil³¹⁻³⁵. Muguda *et al.*^{36,37} proved through the direct tensile test that all XG-treated soil has higher tensile strengths than untreated soil at 7 days. Soldo *et al.*³⁸ carried out splitting tests and showed that XG had the most significant improvement in tensile strength of soil compared with other biopolymers. However, there are few studies on the influence of XG on the tensile strength of loess. The radial splitting tensile test can measure the tensile strength of soil indirectly. It's selected in this paper because of its quick and straightforward operation^{39,40}.

The microstructure is one of the means to explain mechanical behavior. During drying, the XG enhanced the strength of the soil by coating the surface of the soil particles, improved the permeability by filling the pores, and improved erosion resistance by increasing fluid viscosity⁴¹⁻⁴⁴. In addition, the ability of soil to resist external deformation under stress was enhanced by the cation bridging and hydrogen bonds between the carboxylic acid (-COOH) and hydroxyl (-OH) groups of XG and cations on the surface of particles⁴⁵. At present, the scanning electron microscopy (SEM) test is used chiefly for microscopic analysis, but the mercury intrusion porosimetry (MIP) test is rarely used as a quantitative way to describe the porosity and the pore size distribution of soil.

As the basis of the project of the Sanmenxia section of national Highway G310, a series of splitting tension tests based on particle image velocimetry (PIV) test system with different initial dry densities (1.55 g/cm³, 1.63 g/cm³, 1.70 g/cm³) and XG content (0.0%, 0.5%, 1.0%, 2.0%) were carried out. In addition, the SEM and MIP tests were used to conduct qualitative and quantitative analysis on the microstructure of XG-treated loess, which further explained the macroscopic behavior of XG-treated loess.

Materials

The loess was selected from the bridge of Dongshang Village, Lingbao City, Sanmenxia, Henan Province. The essential physical characteristics of loess are shown in Table 1. According to the grain size

distribution curve in Figure 1(a), the loess was mainly composed of 77.24% silt, 14.10% clay and 8.66% sandy. It had a coefficient of uniformity (C_u) and a coefficient of curvature (C_c) of 2.57 and 12.43, respectively. Other basic physical parameters are shown in Table 1. According to the engineering classification standard of soil, it can be classified as well-graded silty clay with low plasticity. Further descriptions of loess can be found in Jiang *et al.*⁴⁶.

The Xanthan gum (XG) used in the test is a white to cream-colored powder, as shown in Figure 1(b). It is an extracellular branching polysaccharide produced by the bacteria *Xanthomonas campestris* and its easy availability and economical cost. XG mainly forms viscous hydrogels with a primary structure consisting of two glucose, two mannoses, and one glucuronic acid. Along with the easy solubility in hot and cold water, XG has strong hydrophilicity, high pseudo-plasticity and high tolerance towards acid, alkali, salt and heat. It is widely used in food, biomedicine and petroleum exploitation⁴⁷⁻⁴⁹. The parameters of XG are shown in Table 2.

Test apparatus and experimental techniques

Splitting tension test equipment based on particle image velocimetry (PIV) technology.

As shown in Figure 2, the PIV test equipment includes an image capture system and a loading system. The image capture system is made up of CCD high-speed industrial camera, floodlights and Davis 8.0 series software. The loading system is a CMT4000 electronic universal testing machine produced by Mester company in the United States, composed of loading equipment and data acquisition system. The universal testing machine can set parameters according to requirements, automatic constant speed loading. Since the linear variable differential transducer (LVDT) deformation sensor and loading device collect information simultaneously, it can be considered that the deformation of the specimen is coordinated with the load imposed by the loading device^{39,40}.

Microscopic test.

SEM tests can qualitatively reveal the arrangement structure of particles and pores on the surface of the soil by using secondary electron signal imaging. A field emission scanning electron microscope (JSM-7600F) produced by JEOL Company in Japan was used in this study⁴¹. The resolution of SEM is 1.0 nm (15 kV)/1.4 nm (1 kV), the acceleration voltage range is 0.1 kV-30 kV, and the amplification can be 1 million times. MIP is an accurate and effective method to determine the pore size distribution of soil through quantitative analysis. The Autopore-9600 instrument manufactured by Micromeritics was used in the MIP test⁵⁰. Its measurable pore diameter ranges from 5.5 nm to 363.6 μm , and the pressure ranges from 3.4 kPa to 227.5 MPa.

Table 1

Physical parameters of loess

Physical parameters	Values
Liquid limit, w_L (%)	26.92
Plastic limit, w_P (%)	18.06
Plasticity index, I_p	12.79
Specific gravity of the solid, G_s	2.70
Natural water content, w_0 (%)	5.10
Natural dry weight, ρ_{d0} (g/cm ³)	1.33
Optimal water content, w_{opt} (%)	17.70
Maximum dry density, ρ_{dmax} (g/cm ³)	1.74

Table 2

Parameters of XG

Biopolymer	Grade	Viscosity/cP	Pyruvic acid/%	Dry weight loss/%
XG	Food	1475	≥ 1.5	9.0

Specimen preparation and testing program.

In order to realize dehydration, the initial water content of the specimens in all tests was designed to be 20%^{27,28}. The contents of XG were controlled at 0%, 0.5%, 1% and 2% by mass ratio (m_b/m_s). The specimens were prepared by dry mixing technique⁴², which was to mix quantitative XG and loess powder evenly and then add quantitative water according to water content. After that, the XG-loess mixtures were placed in a sealed desiccator for braising for 24h. Specimens (63.5 mm in diameter and 25.2 mm in height) were compacted in a cylindrical shape mold (shown in Figure 3). The curing conditions of specimens were room temperature of 25°C, relative humidity of 50%, and natural air drying.

Splitting tension test.

(1) Firstly, the ring specimens under three initial dry densities of the specimens of 1.55 g/cm^3 , 1.63 g/cm^3 and 1.70 g/cm^3 were prepared and dried for 7 days. (2) The specimens were placed on the universal testing machine, and the position of the upper pressure plate was adjusted until it contacted the top of the specimen. At the same time, the pressure sensor was ensured that it was well connected to the computer, and the collection parameters of the loading device were set according to the test requirements. The constant displacement control was selected, and the displacement descent rate was set to 1.4 mm/min . (3) The floodlights and camera were adjusted to ensure that image sharpness and field of view were at their best. (4) The total number and frequency of photos in the PIV measurement system were set to 1500 and 7 photos/s, respectively. The test should be finished after the apparent failure of the specimen. (5) According to the load-displacement curve, the pictures taken at each stage were selected, and the PIV analysis system was used to compare the pictures. The soil deformation field in the process of crack development was analyzed, and the displacement vector figure was generated.

Microscopic test.

For soils with different initial dry densities, the change law of microstructure is similar^{51,52}. Therefore, the specimens with a dry density of 1.63 g/cm^3 were taken as the representative in the microscopic test. The specimens with 0% and 2% XG contents were prepared and cured for 7 days. To preserve the original structure of the specimens as much as possible, the specimens were placed in liquid nitrogen for rapid cooling and then freeze-dried for 24h. For the next step of the tests, the specimens were cut into small pieces. Before SEM observation, a thin layer of gold was plated on the surface of the specimens. Multiple soil points were taken for shooting, and the magnifications of SEM images were 50, 100, 200, 500, 1000, 2000, and 10000, respectively. To ensure the integrity of the specimens, two parallel tests were carried out for each group of soil specimens, and the best specimen was selected for subsequent SEM and MIP tests.

Results And Discussion

Microscopic test.

SEM images. The SEM images are used to describe the microstructure with different XG contents. The SEM images magnified by 500 and 1000 times of soils with 0% XG content are illustrated in Figure 4(a)-(b). After 7 days of dry curing under constant temperature and humidity, the full development of aggregated structure made the particles or aggregates appear angular. The arrangement of particles was mainly mosaic, and the adhesion only came from the connection of the particles with point-point contact and face-point contact.

The SEM images magnified by 500 times of soils with 2% XG content are depicted in Figure 4(c). Compared with the images for untreated soil, the use of the XG induced the formation of flocs and made soil particles contact each other by lines or faces³⁵. As the image enlarges to Figure 4(d), the cross-linking between XG and soil can be further observed. After 7 days of dry curing, XG became short and agglomerated in the process of precipitation and shrinkage, and the viscosity of XG itself increased³². The XG initially filled in or near the pores either developed into bonding surface, merged into bridge links, or accumulated into fiber matrices, which connected the adjacent soil frameworks closely^{23,25,26,29,42}. As the contact area among the soil particles increased, the integrity of soil particles was enhanced, and the particle size of soil skeleton particles became larger.

MIP images. Figure 5(a)-(b) depict the cumulative mercury intrusion curve and pore size distribution (PSD) curve of specimens with 0% and 2% XG contents, respectively. As can be seen from Figure 5(a), the pore diameter of the specimens with 0% and 2% XG contents were mainly concentrated in the pore diameter range of 1-30 μm . The pore volumes of the two types of specimens were almost the same.

As presented in Figure 5(b), the PSD curves of two types of specimens showed multi-peak characteristics. According to the previous microstructural change tests results^{53,54}, the pores of loess specimens were divided into ultra-micropores ($< 1 \mu\text{m}$), micropores (1-5 μm), mesopores (5-45 μm) and macropores ($\geq 45 \mu\text{m}$). The PSD curves of untreated and XG-treated specimens were the same in the range of ultra-micropores and macropores. However, they had different performances in the concentrated distribution of micropores and mesopores. With the addition of XG, the pore size corresponding to the peak intensity of PSD in the ultra-micropores ranges and its peak intensity remained unchanged, which were 0.04 μm and 0.4 mL/g, respectively. The pore size corresponding to the peak intensity of PSD in the micropores ranges remained unchanged, both 3.4 μm , and the peak intensity increased from 0.14 mL/g to 0.20 mL/g. The pore size corresponding to the peak of PSD in the mesopores ranges decreased from 17.3 μm to 7.3 μm , and the peak intensity increased from 0.13 mL/g to 0.18 mL/g. In the drying curing process, the soil particles and XG of the XG-treated specimen fully shrink and aggregate. Therefore, the XG-treated specimens are dominated by micropores and mesopores. The peak values of XG-treated specimens were higher than that of untreated specimens.

Splitting tension test.

Load-displacement curve and splitting tensile strength. The load-displacement curves of the XG-treated specimens with initial dry densities of 1.55 g/cm³, 1.63 g/cm³ and 1.70 g/cm³ are described in Figure 6(a)-(c). The marks below the feature points represent XG content. After curing for 7 days, the load-displacement curves of specimens with different XG contents showed a prominent strain-softening type. These curves could be divided into different stages: OA: The slope of load-displacement curves increased gradually, and the stress concentration occurred at the contact position between the upper and lower pressure head and the specimen; AB: The load of the specimen increased linearly with the increase of

displacement. At this stage, the specimen deformed in elasticity, and the interior of the specimens tended to be compact under stress transfer; BC: The curves dropped sharply after reaching the peak value. The specimen entered the stage of instability failure, and the vertical cracks on the specimen surface began to appear clearly. CD: The load of the specimen was stable with the increase of displacement. The cracks developed on the surface and inside of the specimen until the specimen was utterly destroyed^{39,40}. With the rise in XG content, the softening phenomenon of the load-displacement curve was more pronounced, and the brittleness of the specimen was greater. The load-displacement curves of different initial dry densities varied with XG content. In general, the higher the dry density, the more prominent the softening phenomenon, the greater the failure load.

The splitting tensile strength (σ_t) is derived from the failure load (P) and the diameter (d) and height (t) of the specimen, as shown in Equation 1. The P is the load corresponding to point B in the load-displacement curve. It can be seen from the summary table of splitting test data (Table 3) that the water content (w) and saturation (S_r) of the specimens increased slightly with the increase of XG content. As shown in the relationship curves between splitting tensile strength and XG content with different initial dry densities (Figure 7), all XG-treated specimens showed higher splitting tensile strength than untreated specimens^{36,37}. The higher the XG content and dry density, the greater the splitting tensile strength of the specimen. The splitting tensile strength of specimens with 2% XG content was the highest. Under the dry density of 1.55 g/cm³, 1.63 g/cm³ and 1.70 g/cm³, the strength of soil could be increased by 6.58 times, 5.62 times and 6.06 times, respectively. After curing for 7 days, the water content of the specimens was lower than 2%, and the specimens were in a dehydrated state, which provided enough conditions for the formation of the XG matrix and the mutual bonding between the XG and soil particles²⁶. The dense fiber network formed by XG could reduce the loss of water, thereby increasing the water content and saturation. The ability of the soil to resist external forces mainly came from the bond strength of the biopolymer. Having bridge connection or dense mesh formed by XG made the stronger interparticle connections between soil and XG so that the splitting tensile strength was enhanced during the dehydration process^{32,35,41}.

$$\sigma_t = -\frac{2P}{\pi dt} \quad (1)$$

Table 3

Summary of splitting tension tests data.

$\rho_{d0}/(\text{g}/\text{cm}^3)$	$(m_b/m_s)/\%$	$w/\%$	$S_r/\%$	e	α_t/kPa	Fracture type
1.55	0.0	1.04	4.10	0.69	75.55	I
	0.5	1.21	4.70	0.70	224.01	I
	1.0	1.33	5.13	0.70	231.39	I
	2.0	1.50	5.76	0.70	572.96	I
1.63	0.0	1.23	5.21	0.62	111.39	I
	0.5	1.24	5.30	0.63	333.88	I
	1.0	1.31	5.63	0.63	502.69	I
	2.0	1.54	6.59	0.63	737.33	II
1.70	0.0	1.16	5.55	0.56	139.63	I
	0.5	1.18	5.56	0.57	569.56	I
	1.0	1.34	6.33	0.57	644.07	I
	2.0	1.54	7.29	0.57	986.18	II

Fracture propagation and displacement vector field. The fracture propagation and displacement vector fields obtained from the splitting tension test can be divided into two types, as shown in Figure 8. The darker areas in the images have more concentrated stresses, more significant deformations and more noticeable cracks. The displacement vector fields of the two types had the same performance at point B. The specimen at point B had compression deformation without apparent cracks, and the displacement was mainly concentrated in the specimen interior. They were different at point C and D. I: After the load burst drops to point C, a fine crack appeared in the center of the specimen, and the displacement vector field was symmetrically distributed on both sides of the splitting surface, then the specimen began to fail. At point D, the primary cracks were radial vertical, and the secondary fractures were well developed. The displacement vector field obtained by the PIV technique is a blank area for the failure part of the specimen due to its large displacement. II: After the peak value, the specimen did not appear the fine crack as shown in point C, but immediately appeared the penetrating cracks when the load burst dropped to point D and then fell directly. The splitting types of specimens with different initial dry densities are also summarized in Table 3. The brittle failure and radial vertical crack of specimen were consistent with the apparent strain-softening of the load-displacement curve. No matter the dry density, the brittleness of loess was effectively increased by XG treatment over those of pure loess after curing for 7 days. The specimens presented as class II had a higher XG content (2%) and larger dry density (1.63 g/cm³, 1.70 g/cm³). The increase of brittleness of the specimen was attributed to the stronger cementation and

denser structure formed by the cross-linking between XG and soils. When XG content was 2%, the brittleness of the specimen was the highest.

Conclusions

Based on the particle image velocimetry technique, a series of splitting tensile tests were carried out on XG-treated loess with different XG contents and initial dry densities. The microstructure of the specimens was qualitatively and quantitatively analyzed by scanning electron microscopy and mercury intrusion porosimetry. The main conclusions drawn from the study are as follows.

In the process of drying curing, the XG gradually shrunk and accumulated. They either developed into bonding surfaces, merged into bridge links, or accumulated into fiber matrices, forming a dense network closely connecting the adjacent soil frameworks. The micropores and mesopores mainly existed at the specimens treated with xanthan gum. The corresponding peak values of the PSD curve of xanthan gum-treated specimens were higher than those of untreated specimens.

The XG has been found to be effective for the stabilization of loess. After drying for 7 days, the load-displacement curves of all the specimens were shown strain-softening type. The splitting tensile strength of the specimens increased nonlinearly with the increase of XG content under different initial dry densities. The addition of the XG could increase the splitting tensile strength of loess by about 6 times. The specimens with higher XG content had a larger water content and saturation. The fiber interconnected network formed by XG could not only prevent the loss of water but also enhance the splitting tensile strength.

In the splitting tensile test, the fracture propagation and displacement vector field had an apparent one-to-one correspondence with load-displacement curve in stage division. Based on the PIV technology, the compression deformation stage, initial cracking stage, crack development gradually until the penetrating failure stage could be observed. The specimens presented radial vertical primary crack and relatively developed secondary crack. With the increase of XG content and dry density, the soil structure became denser and more flocculated, leading to the rise in brittleness of specimens. The penetrating cracks appeared directly after the peak splitting tensile strength with the specimens with 2% XG content and the dry density of 1.63 g/cm^3 and 1.70 g/cm^3 .

Declarations

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Author contributions

Jiang. Tong and Jun-ran Zhang initiated the study. Jun-ran Zhang and Jin-di Zhao designed the scope of study. Jin-di Zhao conducted the experiments and wrote the manuscript. Jun-ran Zhang reviewed the manuscript.

Competing interests

The authors declare no competing interests.

Additional information

Correspondence and requests for materials should be addressed to J.r.-Z.

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Figures

Figure 1

(a) Particle-size distribution of loess, (b) XG powder.

Figure 2

Schematic diagram of splitting tension device

Figure 3

Schematic diagram of split tension test specimens.

Figure 4

(a) SEM image of the untreated soil at 500×, (b) SEM image of the untreated soil at 1000×, (c) SEM image of the 2% XG-treated soil at 500×, (d) SEM image of the 2% XG-treated soil at 1000×.

Figure 5

MIP test results. (a) Cumulative intrusion curve, (b) Pore size distribution density curve.

Figure 6

Load-displacement curves of specimens with different initial dry densities. **(a)** 1.55 g/cm³, **(b)** 1.63 g/cm³, **(c)** 1.70 g/cm³.

Figure 7

Relationship between splitting tensile strength and XG content.

Figure 8

Fracture propagation and displacement vector field of specimens. **(a)** I, **(b)** II-1.63 g/cm³, **(c)** II-1.70 g/cm³.