A Novel Clean Biopolymer-Based Additive To Improve Mechanical And Microstructural Properties of Clayey Soil

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Research Article

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Abstract

Conventional soil additives, as a sector of construction materials, can cause serious environmental problems due to their cement-dependent nature. To eliminate destroying effects of these kinds of additives, environmentally friendly ones have gained great consideration. Although polysaccharides are considered as powerful biocompatible soil additives in recent studies, the effect of their crosslinked hydrogel with elevated rheological and adhesive properties has been very rarely investigated in soil stabilization. In this paper, calcium chloride was used as ionic crosslinker to enhance recently proposed Persian gum biopolymer as a novel polysaccharide-based additive for clayey soil stabilization. A group of macro and micro scale tests including unconfined compressive strength (UCS), direct shear test (DS), freeze thaw durability, scanning electron microscopy (SEM), stereo zoom microscopy (SZM), Brunauer, Emmett and Teller (N2-BET) and X-ray diffraction (XRD) were conducted. The tests were also performed on soil treated with xanthan gum as a common hydrocolloid of soil stabilization. As the results show, the introduced ionic crosslinked hydrogel at its optimum content has superior performance than well-known xanthan gum in terms of soil strength and ductility. Crosslinking has also positive effects on durability results of pure Persian gum to make it comparable to xanthan gum. Consumption of lower amounts of Persian gum at presence of ionic crosslinker can make stabilization projects more economical efficient. Microscale tests also confirmed the powerful impact of optimum modified hydrogel on soil interstructure by filling the pores, agglomeration of soil particles and formation of new cementitious compounds in the mineralogical structure of the soil.

Introduction

Non-traditional additives of soil stabilization such as resins, polymers and geopolymers, acids, ions and nanomaterials (Ahmadi et al., 2020; Ghadir and Ranjbar, 2018; H. Ghasemzadeh et al., 2020; Hosseini et al., 2019; Latifi et al., 2016; Mehrpajouh et al., 2021; Mirzababaei et al., 2017) have been developed to eliminate detrimental effects of conventional ones like bituminous material, cement, gypsum, lime, fly ash and blast furnace slag on the environment (Khemissa and Mahamedi 2014; Sharma and Sivapullaiah 2016; Ghasemzadeh and Tabaiyan 2017; Baldovino et al. 2019; James 2020) including energy and natural resources consumption, high greenhouse gas emission in their production procedure, increase of soil pH after stabilization and destruction of plant cover and groundwater quality. Although these non-traditional additives had effective role in air emission control, energy conservation and environmental protection, they were not completely bio compatible and renewable. With the increasing attention to the environmental issues and in order to eliminate any stress on the environment, some environmentally friendly additives like microbial induced calcite precipitation, fungus, enzymes, and biopolymers such as lignin, natural gums, chitosan, casein and sodium caseinate (Cabalar et al. 2017; Fatehi et al. 2018; Kushwaha et al. 2018; Sharaky et al. 2018; Wen et al. 2018; Bu et al. 2019; Lim et al. 2020) have been introduced to soil stabilization. Natural gums, also called Hydrocolloids, are long chain polymers with high amounts of hydroxyl groups in their structure that cause strong affinity to water molecules and make them hydrophilic colloids or “hydrocolloids”. Considerable thickening, film forming, viscosity
enhancement and adhesive properties of natural gums have made them as applicable materials in different industries such as food, tissue and medicine (Pereira et al. 2018; Mohammadinejad et al. 2019). Such properties also put them in the center of attention for soil investigations. Xanthan with microbial source, guar, taragacanth and ghatti with plant origin, sodium alginate and agar as sea weed gums are examples of natural gums' application in soil environment with the purpose of erosion reduction and mechanical behavior improvement (Ayeldeen et al. 2017; Cabalar et al. 2018; Arab et al. 2019; Cheng and Shahin 2019; Dehghan et al. 2019; Biju and Armpalli 2020; Anandha Kumar et al. 2021). The enhanced mechanical and physical properties of the soil caused by hydrocolloids on one hand and the increasing global need for renewable and green materials on the other hand have encouraged investigators to seek new sources of hydrocolloids as soil stabilizers.

Persian gum is a novel source of hydrocolloid biopolymers categorized as plant exudates gums and obtained from branches of wild almond (Amygdalus scoparia) plants in arid and semi-arid regions of Zagros forest in Fars province, Iran (Dabestani and Yeganehzad 2019). This polysaccharide has found wide applications in various industries such as food, pharmacy and textile (Abbasi and Rahimi 2015; Chahibakhsh et al. 2019; Raeisi et al. 2019) due to its comparable effects with commonly used hydrocolloids (Amini Rastabi and Nasirpour 2017; Dabestani et al. 2018; Raoufi et al. 2019). In a recent paper, Ghasemzadeh and Modiri (Ghasemzadeh and Modiri 2020) introduced Persian gum as a novel kind of hydrocolloid soil stabilizers. The study proved comparable soil strengthening capabilities of this gum to well-known xanthan and guar gums at their optimum contents. However, improving properties of Persian gum through modification methods can be a major step forward to make it a more enhanced soil stabilizer.

Modification of polysaccharides is changing their molecular structure by strengthening bonds and interactions between polymer chains to obtain networks with more enhanced viscosity, solubility and rheological properties (Akhtar and Ding 2017). The obtained hydrogels have rigid structure that trap water within them and resist against flow and shear forces (Saha and Bhattacharya 2010). Typically, the modification methods are categorized into chemical and physical crosslinking (Patil and Jadge 2008). In physical crosslinking, physical interactions between polymer chains are formed. However, in chemical crosslinking, covalent bonds between different polymer chains are induced. Among modification methods, physical ones with less chemical contamination risk (due to the absence of crosslinking agent that makes obtained hydrogels non-toxic and biocompatible) have been preferred by many researchers (Ullah and Chen 2020). The existing methods for physical crosslinking include ionic interaction, crystallization, hydrogen bonds and hydrophobic interactions (Hu et al. 2019). Ionic crosslinking as a way of physical modification methods involves entanglement of ionic moiety with polymer chains through non-covalent interactions to obtain a more tightly bonded network. There are conducted studies on ionic crosslinking of natural gums hydrogels such as xanthan, alginate and Arabic gums that demonstrate the positive effect of ionic crosslinking on physiochemical and rheological properties of these gums (Mbah et al. 2012; Yang et al. 2013; Petri 2015). However, application of crosslinked hydrogels of natural gums in soil environment has been mostly restricted to water maintenance and purification purposes (Masoumi and Ghaemy 2014; Zonatto et al. 2017). To the best of the authors’ knowledge, the only study on ionic
crosslinking of polysaccharides for mechanical improvement of soil is carried out on sodium alginate which showed the effectiveness of Ca-alginate as an ionic crosslinked polysaccharide (Wen et al. 2019). Persian gum with numerous functional groups (Dabestani and Yeganehzad 2019), can easily interact and crosslink with other materials (Mohammadi et al. 2016; Samari-Khalaj and Abbasi 2017). On the other hand, calcium chloride is a practical and inexpensive ionic crosslinker that has the ability to connect polymer chains via $\text{Ca}^{2+}$ ionic moiety and form three-dimensional molecular networks (Khalesi et al. 2012; Yang et al. 2013). Therefore, it can be an appropriate candidate for crosslinking Persian gum hydrogels.

As an extension of the previous study (Ghasemzadeh and Modiri 2020), this paper is an attempt to modify Persian gum stabilizing capabilities by physical crosslinking. For this reason, the effect of crosslinked hydrogel of Persian gum using calcium chloride on low-plasticity clay, as a kind of problematic soil, has been investigated. To understand how enhanced viscosity and rheological properties of modified hydrogel of Persian gum affect stabilization goals, mechanical strength and freeze thaw durability were used as soil improvement indicators. The influential factors including PG content, crosslinker concentration, moisture content and curing time were studied. Also, some micro-scale tests including scanning electron microscopy (SEM), stereo zoom microscopy (SZM), $\text{N}_2$-based Brunauer, Emmett, and Teller ($\text{N}_2$-BET) test and X-ray diffraction (XRD) analysis were conducted to provide insight into underlying mechanism of soil strengthening before and after stabilization at micro level.

**Materials**

**2.1. Soil**

The used soil is white kaolinite, sourced from Sahand region of Azerbaijan province, Iran. It was provided from Khak-Chini Company in 35 kg bags in white powder form. The used cohesive fine-grained soil has liquid and plastic limit of 45 and 25.7, respectively and is classified as low-plasticity clay (CL) according to the Unified Soil Classification (ASTM D2487). Compaction characteristics of the used kaolinite include maximum dry density of 1.925gr/cm$^3$ and optimum moisture content of 25% (ASTM D1557). The kaolinite soil with the presented properties (Table 1) was chosen considering its importance as a kind of commonly used problematic soil.
Table 1
Soil properties

| Soil properties                             | Standard    | Values |
|---------------------------------------------|-------------|--------|
| Unified soil classification system          | ASTM D2487  | CL     |
| Fine percentage, P#200 (%)                  | ASTM D854   | 82     |
| Plasticity index (%)                        | ASTM D4318  | 19.3   |
| Liquid limit (%)                            | ASTM D4318  | 45     |
| Plastic limit (%)                           | ASTM D4318  | 25.7   |
| Maximum dry density (gr/cm3)                | ASTM D1557  | 1.925  |
| Optimum moisture content (%)                | ASTM D1557  | 25     |
| Specific gravity ($S_g$)                    |              | 2.66   |
| Chemical compounds (%)                      | ASTM D8064  |        |
| SiO$_2$                                      |             | 63     |
| Al$_2$O$_3$                                  |             | 24     |
| Fe$_2$O$_3$                                  |             | 0.55   |
| TiO$_2$                                      |             | 0.04   |
| CaO                                          |             | 1.2    |
| Na$_2$O                                      |             | 0.33   |

2.2. Xanthan Gum

Xanthan gum is a high molecular weight hydrocolloid that can be produced from fermentation of carbohydrates by a bacteria called X. campestris (Palaniraj and Jayaraman 2011). The chemical structure of this microbial sourced gum is composed of hexose units including D-glucose, D-mannose, and also D-glucuronic and pyruvic acids. The main chains of β-D-glucose is repeatedly connected to the side chains of trisaccharides (α-D-mannose, β-D-glucuronic acid and β-D-mannose that contains an acetyl group) to form a helical structure. β-D-glucose is (1→4) linked to α-D-mannose in the backbone. Also β-D-mannose is (1→4) linked to β-D-glucuronic acid and β-D-glucuronic acid is (1→2) linked to the α-D-mannose in the side chain. pyruvate and acetyl groups are attached to the terminal β-D-mannose at C6 (Petri 2015) (Fig. 1). The pyruvic units and glucuronic acids are responsible for negative charge of Xanthan. This anionic nature gives some desired characteristics to the gum such as high hydration and water solubility. Xanthan gum, even at low concentrations, can considerably increase the viscosity of liquids and its viscous gel is stable under different pH values and temperatures (Sworn 2009).

2.3. Persian gum
Persian gum, also known as Zedo, Shirazi, Farsi and Angum gum, is a complex polysaccharide classified as plant exudate gums. Despite its similarity to the known Arabic gum, some properties of Persian gum such as small values of protein, existing xylose and mannose groups and the ratio between galactose and arabinose help to distinguish it from its competitor (Dabestani et al. 2018). The arabinogalactan structure of PG and its similar induced physical and rheological properties to conventional hydrocolloids (Dabestani and Yeganehzad 2019), has made it a proper alternative for previously used polysaccharides. According to the 13C-NMR, 1H-NMR and HPLC, backbone of Persian gum includes galactose (1→3 linked β-D-Glap) and rhamnose and side chains contain (1→6) linked β-D-Glap and (1→3) linked α-L-Araf residues (Abbasi and Rahimi 2015; Molaei and Jahanbin 2018) (Fig. 1). The monovalent and divalent cations in the structure of PG including K, Zn, Mg, Na, Fe and Ca constitute major elements of this newly introduced polysaccharide (Abbasi 2017). Persian gum has 30% soluble part which solves in cold water and 70% insoluble part that solves partially in hot water. According to the GC/MS chromatographic method, the main monosaccharides of PG are arabinose and galactose units with 2:1 ratio. Other constituting monosaccharides existed in the structure of PG include mannose, rhamnose and xylose with smaller values (Fadavi et al. 2014). FTIR analysis of this novel gum (Dabestani et al. 2018) revealed presence of -CH₂, -CH₃, O-H, C-H, C-O, C-C, C = O, -COO-, C-OH, alcohol and amide groups. These functional groups, along with considerable molecular weight and branched structural shape of PG, cause high tendency of it to interact with other materials. There are some conducted studies about the feasibility of interactions between Persian gum and other hydrocolloids such as sodium caseinate, taragacanth gum and gelatin (Sadeghi et al. 2018; Khodaei et al. 2020). PG has high stability against environmental conditions such as pH (2 to 11) and heat (up to 90°C) that guarantee its powerful performance in some sensitive industrial systems (Dabestani and Yeganehzad 2019). The used Persian gum was purchased from a mucilage and gum provider company named Reihan Gum Parsian in white powder form. According to the manufacturer, it contains 91.3% carbohydrate, 2.16% total ash, 1.2% protein and 0.2% fat. The physiochemical properties of used biopolymers including xanthan and Persian gum are summarized in Table 2.
Table 2
Physiochemical properties of used biopolymers, xanthan and Persian gum

| property                      | Persian gum | Xanthan Gum |
|-------------------------------|-------------|-------------|
| Appearance                    | white powder| white powder|
| Category                      | plant exudate| microbial   |
| water solubility              | 30%         | 100%        |
| Molecular weight (Da)         | 4.6×10^6    | 5.0×10^6    |
| viscosity for 1% solution (mPa.s) | 48          | 1100        |
| Charge                        | Negative    | Negative    |
| Ash (%)                       | 2.19        | 3.5         |
| Protein (%)                   | 1.2         | 5           |
| Carbohydrate (%)              | 91.3        | 82          |
| Moisture (%)                  | 5.14        | 9.4         |

2.4. Calcium chloride solution

To provide Calcium chloride Persian gum (Ca-PG) hydrogel, Persian gum should be dissolved in calcium chloride (CaCl₂) solution. CaCl₂ solutions were prepared by mixing the required amount of CaCl₂ powder with distilled water at room temperature. CaCl₂ powder was purchased from a local supplier with properties mentioned in Table 3.

Table 3. Physiochemical properties of calcium chloride
| property                              | Values                        |
|--------------------------------------|-------------------------------|
| Appearance                           | A white odorless granule      |
| Soluble/insoluble in water           | 100% soluble                  |
| Density (gr/cm³)                     | 2.15                          |
| Molar mass (gr/mol)                  | 110.98                        |
| pH                                   | 8                             |
| Calcium (%)                          | 38                            |
| Chloride (%)                         | 59.4                          |
| NaCl (%)                             | 1.6                           |
| MgCl₂ (%)                            | 0.09                          |
| Other impurities (%)                 | 0.91                          |

**Sample Preparation**

First, the gravel and large particles were removed from the soil by passing it through a 2 mm sieve. The maximum dry density (MDD) and optimum moisture content (OMC) were calculated from the results of standard proctor test (ASTM D1557) (ASTM D698 2003). To prepare xanthan and Persian gum treated samples, the biopolymer contents of 1, 1.5, 2, 2.5 and 3% by weight of dry soil were dissolved in the required water for optimum moisture content and the obtained solutions were mixed with dry soil. For crosslinked specimens, the aforementioned biopolymer contents were dissolved in CaCl₂ solutions of 0.5, 1, 1.5, 2 and 2.5 molarities and the obtained Ca-PG solutions were combined with the soil and homogenized using hand and palette knives.

For UCS test, the mixtures were compacted in cylindrical molds using a hydraulic jack based on ASTM D2166 (ASTM D2166 2016) to achieve maximum dry density and optimum moisture content. The diameter and height of used molds were 38 and 76 mm, respectively. A hydraulic specimen extruder with hand operating mechanism is used to extrude specimens from the steel molds. The obtained samples were trimmed and cured in a controlled room of about 22°C for 7 days. At least three specimens were constructed for a specified additive content and the represented strength results were the average of tested samples. To conduct DS test, the mixtures were placed in steel cubic-shaped molds of 60×60×20 mm and compacted using static pressure approach to achieve optimum moisture content and maximum dry density. The samples were then extruded using a hand operated jack and air dried in a controlled room of about 22°C for 7, 14 and 28 days. For durability test, the samples were constructed in accordance with ASTM D558 test method (ASTM 2011) for soil material passing a No. 4 sieve. The soil, additive and water mixture were compacted in a mold with an internal diameter of 101.6 mm and volume of 944 cm³ to reach maximum dry density. The compaction of soil was conducted in three layers by
applying 25 blows on each layer using a rammer from a height of about 30 cm. After removing the molds, obtained specimens were trimmed with knife and cured at the room temperature of 22°C for 28 days.

Small pieces of crushed specimens at the end of UCS test were used to conduct further microscale analysis using SEM, SZM, BET and XRD. Since being dried is the requirement for microscale tests, they were dried in the oven before conducting tests. A very tiny piece of specimen was coated with gold cover before being set into the SEM device. SZM images were taken from cross section of dried UCS specimens. The tiny pieces of specimens were ground and changed to the powder form to conduct BET and XRD tests. Degassing of specimens is the prerequisite for conducting BET tests. For this purpose, the samples were exposed to the nitrogen gas injection in a glass cell with high vacuum temperature of 130°C for 3 hours. The powder specimens were processed in the sample container for BET and XRD tests.

The designated labeling to identify specimens has five parts: The abbreviation of K is used as the first part to represent kaolinite soil. The second part includes UNT for pure soil and hydrocolloid name for treated samples: PG for Persian gum and XG for xanthan gum. The third part demonstrates PG content. The fourth part, C, shows the CaCl$_2$ crosslinking agent and the fifth part is related to its molar concentration. For example, KPG2C2 is related to a kaolinite specimen stabilized with Ca-PG hydrogel, in which 2% PG by weight of dry soil is dissolved in 2 molar CaCl$_2$ solution.

**Testing Program**

Samples with different contents of biopolymer and crosslinking agent were examined through UCS tests according to the ASTM D2166 (ASTM D2166 2016). The samples were put in an automated compression machine equipped with a data acquisition system, which is able to record applied load and axial deformation. The strain rate is considered to be 1% per minute. The peaks of stress-strain curves demonstrate the maximum compressive strengths that can be endured by the specimens.

Shear parameters of the soil were evaluated through the results of DS test according to the ASTM D3080 (ASTM D3080 2020). Each cured cubic specimen was placed in the shear box and a strain rate of 0.8 mm/min was applied on it until the sample failed or experienced maximum horizontal displacement of 10 mm (Latifi et al. 2015). The test was conducted on each sample applying three normal stresses including 100, 200 and 300 kPa. Cohesion and friction angle for each specimen were estimated considering obtained failure envelopes.

Durability test of specimens against freeze thaw cycles was conducted according to the ASTM D560 (Standard 2016). The 28 days cured specimens were placed in the moist room for 7 days. After that they were exposed to freeze thaw cycles. Each freeze thaw cycle includes placing the samples in a freezing cabinet (having a temperature of -23°C) for 24 h and then storing them into a moist room with 23°C temperature and relative humidity of 100% for 23h. The procedure was continued for 12 cycles. The mass and moisture loss at the end of each cycle were recorded.
SEM is a common approach for visualizing micro-structure of the materials. In this study, SEM images of pure and treated specimens for optimum content of pure and crosslinked Persian gum were prepared to investigate how modified hydrogel of Persian gum interacts with soil particles.

SZM is the other advantageous imaging method that uses reflected light of the materials to prepare images from their surface. The samples were placed at Motic SZM-140-143-FBGG Stereo Zoom Microscope with considerable zoom property that enables it to prepare high quality images with comprehensive details.

$N_2$ based Brunauer-Emmett-Teller is a powerful test for determination of pore volume and specific surface area information. To conduct BET test, Nitrogen gas was pumped into the sample container to make reaction with the solid surface of particle. Considering gas adsorption model and adopting BET equation, surface area of particles can be estimated. Pore volume is also recognized considering the volume of the adsorbed nitrogen gas at 77°K and 1atm.

The effect of additive on the mineralogical structure of soil can be distinguished from the variations in the XRD patterns of untreated and treated soils. Formation of some new compounds in soil structure can illustrate the macroscale enhancement of soil mechanical features. The XRD tests were conducted using X’pert pro MPD diffractometer made by Malvern Panalytical. Radiation of Cu-Kα at 2θ range of 0.945° to 99.953° with $\lambda = 1.54A°$ and loading step size and time of 0.026° and 2 s was employed. XRD data analysis was conducted through Eva valuation software of Bruker company using its comprehensive database of known compounds.

Results And Discussion

5.1. Unconfined compressive strength test

The UCS values of untreated and treated soil specimens with xanthan gum, Persian gum and crosslinked Persian gum are shown in Fig. 2. The biopolymer contents of 1, 1.5, 2, 2.5 and 3% by weight of dry soil were used to treat the soil. For crosslinked specimens, the aforementioned contents of PG were dissolved in CaCl$_2$ solutions of 0.5, 1, 1.5 and 2 molarities. The goal is to determine the proper combination of Ca$^{2+}$ ions and PG to improve viscosity and rheological properties of formed hydrogels. As it can be seen, for each specified CaCl$_2$ molarity, an increasing and decreasing trend is observable in UCS results as PG content grows. The amount of additive that causes maximum compressive strength is known as its optimum content. The obtained optimum additive contents are 1.5% for xanthan gum, 2.5% for pure PG treated specimen and 1, 1, 1.5, 2 and 2 for Ca-PG treated ones in the presence of 0.5, 1, 1.5, 2 and 2.5 molarities of CaCl$_2$, respectively. As the results show, the UCS values of xanthan and pure PG treated soil at their optimum content have been increased to 83.5 and 128.3% in comparison to the untreated soil, while for KPG1C0.5, KPG1C1, KPG1.5C1.5, KPG2C2 and KPG2C2.5 specimens, the increase values are 81.3, 98.6, 163, 201.1 and 181.6%, respectively. As can be seen, the most elevated strength result is obtained for the specimen with 2% PG and 2 molar CaCl$_2$. The reason is related to proper content of PG.
and calcium chloride that causes effective ionic crosslinking between polymer chains. The branched structure and functional groups of PG such as C-H, -CH₂, -CH₃, C-C, -COO⁻, C = O, C-O, O-H, C-C and -COOH (Dabestani et al. 2018), also enable it to interact with other materials easily. In addition to polymer chains, Ca²⁺ free stabilizer ions interact with Si and Al of the soil and form improved textural characteristics. However, the low content of Ca²⁺ has no crosslinking role for higher PG contents and causes less contracted interchain reactions which conversely weaken the viscosity of formed hydrogels and mechanical strength results. For instance, the presence of 0.5 molar calcium chloride causes the strength results of 1.5, 2, 2.5 and 3% PG to be reduced and 1 molar CaCl₂ has decreasing effect on the strength of 2, 2.5 and 3% PG. To explain this, one should consider that viscosity of hydrogel as the substantial factor in strength results is highly affected by the content of adding salt due to the exudates gums' polyelectrolyte property. According to the previous studies, when the ratio between crosslinker molarity and gum concentration is very low (less that about 0.5), the least contraction of macromolecules results in reduction of viscosity and binding property of gels (Koocheki et al. 2009), and less UCS results are expected. However, these low concentration of CaCl₂ (0.5 molar) effectively promotes UCS of low PG content (1%) treated sample. Therefore, the relation between ionic solution and PG content is the influential factor that determines whether crosslinking has been occurred or not. Crosslinking has also positive effects on the strain results. The stress-strain curves of optimum crosslinked specimens show more ductility in comparison to xanthan and pure Persian gum at their optimum state.

Reduction in gum concentration using crosslinker agent, is one of the considerable advantages of the crosslinked hydrogels. As it can be seen in Fig. 2, the strength improvement caused by 1.5% xanthan gum can be achieved using 1% of PG at presence of 1.5 and 1 molar calcium chloride. Also, the required pure PG content to achieve maximum UCS can be reduced from 2.5% to less than 1.5% (the optimum content of xanthan gum) in the presence of 1 molar CaCl₂ solution. Since the preparation process of the gums to make them applicable in industry is sometimes complicated and expensive, the restricted consumption of them in the soil projects can make them economically efficient. Therefore, substitution of some content of PG with CaCl₂ solution as an inexpensive and simple crosslinker that is also environmentally friendly with no dependency to cement industries is recommended.

5.2. Direct shear test

Shear performance of treated specimens with optimum additive contents, obtained from UCS test results, including KXG1.5, KPG2.5, KPG1C0.5, KPG1C1, KPG1.5C1.5, KPG2C2 and KPG2C2.5 were evaluated through direct shear test. The samples were air-dried at 22°C room temperature for curing times of 7, 14 and 28 days. The obtained shear parameters have been shown in Fig. 3. As it can be seen, cohesion of 28 days treated KXG1.5, KPG2.5, KPG1C0.5, KPG1C1, KPG1.5C1.5, KPG2C2 and KPG2C2.5 specimens has been improved to about 142.9, 133.3, 110.8, 120.8, 153.3, 170.8 and 125.8% in comparison to the untreated soil, respectively. The reason is related to the formed agglomerated particles that counteract lots of transitional and rotational stresses in grain scale and improve shear resistance in macro scale. Similar to UCS test results, combination of 2% PG and of 2 molar CaCl₂ solution is the optimum ratio of these two additives that causes the most enhanced shear performance by creation of some extra bonds
between polymer chains. These new bonds have made interstructure of PG hydrogels powerful networks that resist against shear forces. Xanthan and pure PG treated specimens have shown close strength results after 7, 14 and 28 days curing. However, the cohesion parameter of Ca-PG samples has indicated considerable growth over time. This is related to the formation of more bonds in the interstructure of crosslinked hydrogels that are being completed over time. Remarkable strength growth of Ca-PG specimens over time can make them desirable for long term strength achievement objectives. The reduced cohesion parameter for 7 days Ca-PG treated samples in comparison to pure optimum PG treated ones is related to the moisture maintenance property of Ca-PG hydrogels that postpones evaporation of trapped water existed in the hydrogel networks. According to the gel features (Chen et al. 2019), this trapped water prevents the real adhesive properties of gel to be appeared.

As discussed in the previous section, the interesting point about using Ca-PG treated samples is the possibility of PG content reduction to achieve the same strength results. For instance, cohesion parameter of 14 days treated KPG2.5 and KPG1.5C1.5 specimens are approximately the same. The variations of friction angle as the other shear parameter have been shown in Fig. 3 (b). In fact, formed hydrogels play as lubricant in soil biopolymer medium and cause soil particles to move more smoothly on each other. This has led to reduction of internal friction angle due to the stabilizing agent. Similar to the cohesion results, the friction angle value of pure PG treated specimens has no significant variation for curing times of 7, 14 and 28. However, for Ca-PG treated samples, the value of this parameter has shown observable decreasing trend as time passes. The samples with 2% PG and 2 molar calcium chloride showed a friction angle of 25.2° and cohesion of 325 kPa at the end of 28 days curing that resulted in shear resistance of 372.05 kPa under normal stress of 100 kPa, while the pure clay with friction angle of 32° and cohesion parameter of 120 kPa represented 182.5 kPa shear strength under the same normal stress. The growth of shear stress (about 104%) for KPG2C2 specimens is more considerable than the strength increase induced by optimum xanthan gum treated specimens (91%). Therefore, optimum content of crosslinked PG indicates more elevated shear performance in comparison to well-known xanthan gum.

5.3. Durability tests

Weathering condition was studied by applying freeze thaw cycles on untreated and treated soil specimens to evaluate their durability. According to the previous studies (Eskişar et al. 2015; Ahmadi et al. 2020), the optimum content of additives leads to the increase in freeze thaw durability performance of fine grained soils. Therefore, KUNT, KXG1.5, KPG2.5 and KPG2C2 specimens were chosen to be tested. The 28 days cured specimens were retained in a moist room for 7 days and then subjected to the freeze thaw cycles according to ASTM D560 (Standard 2016). The mass and moisture loss of the specimens at the end of each cycle were presented in Fig. 4. As the results show, the amount of mass loss after 12 freeze thaw cycles is 24.1% for untreated specimen, while for the xanthan, pure and crosslinked PG specimens, the mass loss values are 8.28, 19.1 and 10.25%, respectively. The enhancement of soil durability in term of mass loss reduction for treated soils can be explained by the sticky nature of formed hydrogels between soil particles, that binds them together and forms agglomerated particles with
enhanced freeze thaw resistance. Xanthan gum with powerful gel formation property shows the most powerful performance in durability improvement of the soil. The higher mass loss of PG treated soil in comparison to xanthan treated one can be illustrated by the fact that some extent of PG does not contribute in gel formation process due to its insoluble part (Abbasi 2017). Therefore, the gel network of PG is not as strong as xanthan gum in terms of durability improvement. However, the mass loss values of crosslinked specimens are more remarkable than the pure PG treated sample. This shows the success of crosslinking in durability improvement of PG treated samples to reach the mass loss values of xanthan gum treated specimens. The reason is related to the certain amounts of Ca\(^{2+}\) ions that improve viscosity and rheological properties of PG hydrogels by crosslinking polymer chains together.

The same trend has been observed for moisture loss of the specimens during freeze thaw cycles (Fig. 4(b)). The amount of moisture loss after 12 freeze thaw cycles for KUNT, KXG1.5, KPG2.5 and KPG2C2 specimens is 15, 6 and 9.7 and 7%. As represented, the similar durability results of xanthan gum treated soil are accessible using crosslinked hydrogels of PG. The amount of moisture loss for Ca-PG specimens is much lower than pure PG treated soil samples. This is related to the moisture maintenance property of crosslinked hydrogels due to their three dimensional network structure. The decreasing rates of mass and moisture loss at higher cycles for both treated soils demonstrate positive effect of treatment on the durability of soil specimens. The reduction of slope at higher cycles is more obvious for KXG1.5 and KPG2C2 that shows powerful durability performance of xanthan and modified PG treated sample in comparison to pure PG treated one.

5.4. Microscale Test Results

5.4.1. SEM and SZM tests

The microstructures of optimum pure and crosslinked PG treated specimens were compared to the untreated soil in order to discover how CaCl\(_2\) solution affects interparticle interactions at microscale. For this purpose, SEM and SZM microscopic images of KUNT, KPG2.5 and KPG2C2 are shown in Fig. 5. As it can be seen, optimum Ca-PG solution has made the soil interstructure more enhanced and uniform in comparison to optimum pure PG solution and has powerful performance in pore filling of the pure clay. The proper quantity of Ca and PG in KPG2C2 specimens required for powerful crosslinking of polymer chains is the reason for such an enhanced microstructure. The compact microstructure of KPG2C2 verifies the results of UCS tests that represent its superior strength performance. To more quantitatively investigate the effect of pore filling of the crosslinked specimens, image processing was carried out on the SEM images. To separate pores from the soil particles, thresholding was employed using the Huang and Wang (Huang and Wang 1995) method. After segmentation of Pores and soil particles, their areas were calculated. Figure 6 shows the schematic diagram of the employed image processing method. In this figure, pores and soil particles are shown in black and white colors, respectively. According to the segmentation results (Table 4), the treated specimens have shown less void ratio in comparison to the pure soil. KPG2C2 has shown the most reduction (about 33%) in void area.
Table 4. Image processing results of SEM images

|                  | KUNT  | KPG2.5 | KPG2C2 |
|------------------|-------|--------|--------|
| soil area (μm²)  | 28168 | 38228  | 45957  |
| void area (μm²)  | 52740 | 42681  | 34952  |
| void ratio (%)    | 65.2  | 52.8   | 43.2   |

5.4.2. Brunauer, Emmett and Teller (N2-BET) tests

Nitrogen-based Brunauer, Emmett and Teller test as a powerful method for determination of specific surface area and pore diameter, volume and distribution was used. Calculation method of Barret, Joyner and Hallenda (BJH) (Barrett et al. 1951) was used for determination of pore diameters. The tested specimens are pure soil and treated samples at their optimum state. The obtained parameters including BET surface area, BJH average pore diameter and BJH pore volume are represented in Table 5. As the results show, the specific surface area was reduced from 8.17 m²/gr for pure soil to 4.8, 4.64 and 3.95 m²/gr for KXG1.5, KPG2.5 and KPG2C2 specimens. The reduction in specific surface area of the soil particles after stabilization is related to their agglomeration that results in formation of larger particles with reduced specific surface area. Among the treated specimens, the one stabilized with 2 molar CaCl₂ and 2% PG content has superior performance in reduction of specific surface area and particle accumulation. This feature makes it beneficial for soil improvement goals. Pore volume distribution plots of treated and untreated specimens are shown via BJH pore volume diagram at the range of 1–95 nm pore diameter (Fig. 7). The decreasing effect of treatment on pore volume confirms formation of larger particles that fill the pores. As the results show, the optimum content of PG has elevated performance in pore filling of the soil in comparison to optimum xanthan gum. This is related to the higher amount of biopolymer in the optimum state of PG treated specimens (2.5%) in comparison to xanthan gum treated ones (1.5%). Also, combination of 2 molar CaCl₂ and 2% PG caused the most considerable reduction in pore volume of the soil. This confirms formation of more hydrogels and ionic bonds in the interstructure of the crosslinked hydrogel treated soil in comparison to other kinds of treated samples. In addition to strength improvement objectives, the compact interstructure of this kind of soil also makes it a more attractive alternative for permeability reduction purposes.
Table 5
BET test results of pure kaolinite (KUNT) and treated ones by optimum xanthan gum (KXG1.5), Persian gum (KPG2.5) and crosslinked Persian gum (KPG2C2)

| Sample name | BET surface area (m²/gr) | BJH pore volume (cm³/gr) | BJH average pore diameter (nm) |
|-------------|--------------------------|--------------------------|-----------------------------|
| KUNT        | 8.17                     | 0.054                    | 26.45                       |
| KXG1.5      | 4.8                      | 0.047                    | 28.41                       |
| KPG2.5      | 4.64                     | 0.042                    | 31.52                       |
| KPG2C2      | 3.95                     | 0.031                    | 33.43                       |

5.4.3. X-ray diffraction

To realize how stabilizing agent alters the mineralogical structure of the soil, XRD tests were applied on pure and treated soils with optimum additive contents. The XRD patterns of tested samples including KUNT, KXG1.5, KPG2.5 and KPG2C2 are shown in Fig. 8. Pure soil has shown kaolinite peaks at 2θ of 12, 20, 25, 38.5 and 62°, Quartz diffraction lines at 2θ angles of 21, 26.5, 50 and 60° and very tiny trace of calcite and montmorillonite at about 29.5°. In the stabilization process using xanthan and Persian gum, no new peaks can be observed due to the organic nature and very low content of additives that make incomplete reactions with alumina and silica of the soil. However, for KPG2C2 specimens some noticeable peaks were appeared in the XRD spectrum and a slight reduction in some peaks of pure soil was observed. The new peaks of crosslinked PG treated soil were developed at 2θ of 27–28° and 37–38.5°, which are related to calcium silicate hydrate (CSH) (Latifi et al. 2016; Park et al. 2016; Yong et al. 2019), 2θ of 41°, representing the formation of calcium aluminum hydrate (CAH) (Rao et al. 2009; Yong et al. 2019), and 2θ of around 23–24° and 57°, which shows the existence of crystalline calcium aluminum silicate hydrate (CASH) (C Sekhar and Nayak 2019; Sukmak et al. 2019). The abundance of Ca, Al, Si and O in the mixture of Ca-PG specimens prepares the ground for formation of cementitious CAH, CSH and CASH gels. These pozzolanic products are the results of reaction between alumina and silica ions of soil and calcium ions from CaCl₂ solution and PG structure (Abbasi 2017). The new compounds form a hard skeleton that resist against axial and shear forces properly. The reduced peak intensities of kaolinite and quartz compared to the original soil can be the result of Si dissolution in the process of formation of new cementitious products including CSH, CASH and CAH.

Summery And Conclusion

In this paper, ionic crosslinking was utilized to introduce enhanced hydrogel of Persian gum as a novel green soil additive for clayey soil stabilization. CaCl₂ solution was applied as ionic crosslinker to improve rheological properties of PG hydrogel. For this purpose, different contents of PG were dissolved in various molarities of CaCl₂ solution and the obtained solutions were injected into the soil environment. A comprehensive set of macro and microscale tests were used to evaluate the effectiveness of the crosslinked hydrogels in soil stabilization. To compare the results of the introduced hydrogel with
common hydrocolloids of soil stabilization, the tests were also conducted on well-known xanthan gum treated soil. From the mechanical strength test results, combination of 2% PG and 2 molar CaCl$_2$ solution was determined as the optimum additive content. The increase of strength for optimum Ca-PG specimens with the maximum strength of 555.6 kPa (2.86 times the untreated soil) is more remarkable than the strength growth induced by optimum xanthan gum (1.74 times the pure soil). The results of direct shear test also represented more strength growth for Ca-PG treated specimens (104%) in comparison to xanthan treated specimens (91%) at the end of 28 days drying. The considerable cohesion growth of Ca-PG treated samples over time is related to the water maintenance property of crosslinked specimens that cause them to require more time for water evaporation and strength achievement. This feature can make Ca-PG hydrogels favorable for long term strength achievement objectives. Furthermore, substitution of some extent of PG with CaCl$_2$ to achieve the same strength results, is economically efficient considering the difficulties in PG preparation process. In terms of durability characteristics, the improved durability of Ca-PG treated samples in comparison to pure PG treated soil is due to the more cation bonds in the polymer structure and between polymer chains and soil that help to resist against water and mass loss. The compacted interstructure, reduced pore volume and specific surface area for optimum Ca-PG specimens, observed from SEM, SZM and BET results, verified capability of these kinds of specimens to achieve maximum compressive strengths. Also, formation of new sticky crystalline compounds, shown in the XRD pattern of optimum Ca-PG treated soil, is the other reason for enhanced performance of this green stabilizer.

**Further Research**

The efforts towards physical crosslinking of Persian gum using calcium chloride to make it a more powerful material for soil stabilization were successful. The results of macro and microscale tests proved its effectiveness in terms of mechanical strength and durability improvement, pore filling and compacting interstructure of soil in comparison to pure PG treated soil. However, due to the novelty of this kind of additive, some important aspects of soil stabilization such as consolidation, collapse potential, erosion control and durability against wet dry cycles and also dynamic and large scale behavior of it are still unknown. Using newly developed crosslinking methods is the other further step for extension of this work that is strongly recommended. There are various crosslinking methods of polysaccharides that result in robust and smart hydrogels with more elevated characteristics. Using advanced chemistry knowledge to develop PG hydrogels with enhanced mechanical strength can be a major development in the area of soil stabilization with this novel biopolymer. Also, similar to the other common hydrocolloids that are useful for purification and water maintenance objectives via crosslinking, studying application of crosslinked hydrogel of PG in contaminated and agricultural soil has considerable importance.

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**Figures**

**Figure 1**

Chemical structure of used biopolymers, (a) xanthan gum (Sworn 2009) (b) Persian gum (Abbasi and Rahimi 2015)

**Figure 2**
UCS test results for different biopolymer content (1, 1.5, 2, 2.5 and 3 by weight of dry soil) and CaCl2 solution concentration of (a) 0.0 (b) 0.5 (c) 1.0 (d) 1.5 (e) 2.0 (f) 2.5 molar

Figure 3

DS test results for untreated and treated specimens using xanthan, pure and calcium chloride Persian gum treated specimens at their optimum additive contents: (a) Cohesion (b) Friction angle

Figure 4

Durability test results for untreated and treated specimens using optimum xanthan gum (KXG1.5), pure Persian gum (KPG2.5) and crosslinked Persian gum (KPG2C2): (a) mass loss (b) moisture loss

Figure 5

Microscopic images: (a) SEM (b) SZM for pure kaolinite and treated specimens by optimum pure and crosslinked Persian gum including KPG2.5 and KPG2C2.
Figure 6

Schematic diagram of image processing

Figure 7

BET test results: pore distribution of pure kaolinite (KUNT) and treated ones by optimum xanthan (KXG1.5), Persian gum (KPG2.5) and crosslinked Persian gum (KPG2C2)
Figure 8

XRD results of pure (KUNT) and optimum xanthan gum, pure and crosslinked Persian gum treated specimens (KXG1.5, KPG2.5, KPG2C2)

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