

# Alkaline induced-cation crosslinking biopolymer soil treatment and field implementation for slope surface protection

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**Abstract.** Xanthan gum and starch compound biopolymer (XS), an environmentally friendly soil-binding material produced from natural resources, has been suggested as a slope protection material to enhance soil strength and erosion resistance. Insufficient wet strength and the consequent durability concerns remain, despite XS biopolymer-soil treatment showing high strength and erosion resistance in the dried state, even with a small dosage of soil mass. These concerns need to be solved to improve the field applicability and post-stability of this treatment. This study explored the utilization of an alkaline-based cation crosslinking method using calcium hydroxide and sodium hydroxide to induce non-thermal gelation, resulting in the enhancement of the wet strength and durability of biopolymer-treated soil. Laboratory experiments were conducted to assess the unconfined compressive strength and cyclic wetting-drying durability performance of the treated soil using a selected recipe based on a preliminary gel formation test. The results demonstrated that the uniformity of the gel structure and gelling time varied depending on the ratio of crosslinkers to biopolymer; consequently, the strength of the soil was affected. Subsequently, site soil treated with the recipe, which showed the best performance in indoor assessment, was implemented on the field slope at the bridge abutment via compaction and pressurized spraying methods to assess feasibility in field implementation. Moreover, the variation in surface soil hardness was monitored periodically for one year. Both slopes implemented by the two construction methods showed sufficient stability against detachment and scouring, with a higher soil hardness index than the natural slope for a year.

**Keywords:** biopolymer; calcium hydroxide; crosslinking; field application; slope protection; sodium hydroxide; soil hardness

## 1. Introduction

Recently, numerous studies on sustainable soil stabilization methods have been conducted in response to environmental concerns regarding conventional soil binding materials. Utilizing biopolymers within the soil is one of these sustainable ways for improving the soil. Biopolymers, which are excretory products from living organisms (e.g., microbes, fungi, and plants), have shown remarkable ability to enhance the geotechnical engineering properties of soil, such as strength (Kumara and Sujatha 2020, Soldo *et al.*

2020, Amelian *et al.* 2022), hydraulic conductivity (Kim *et al.* 2019, Lee *et al.* 2021), and erosion resistance (Kwon *et al.* 2020). Among the various biopolymers, xanthan gum and starch compound biopolymers (XS) have attracted considerable interest in recent years owing to their enhanced engineering performance and economic efficiency (Chang *et al.* 2020, Seo *et al.* 2021).

The strengthening effect of the XS biopolymer treatment is mainly attributed to pore fluid modification using a viscous biopolymer hydrogel, which has an electrostatic attraction for charged clay particles (Chang *et al.* 2015, Latifi *et al.* 2017). Particularly, the viscous hydrogel is transformed into a tensile film as the biopolymer-treated soil undergoes a drying process. The compressive and shear strengths of soil are increased by providing artificial cohesion and friction angle increase resulting from agglomeration between coarse grains and the biopolymer-clay matrix (Chang and Cho 2019, Soldo *et al.* 2020). However, biopolymer-treated soil exhibits low strength in a hydrated state (i.e., immediately after mixing) because the tensile strength of the biopolymer hydrogel prior to dehydration is insufficient. Moreover, one of the critical drawbacks of biopolymers is their high water sensitivity, which causes them to disintegrate when in contact with

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water or have impaired mechanical properties due to water absorption and swelling induced by hydrophilicity originating from the carboxyl (-COOH) groups in the biopolymer chain (Chang *et al.* 2016, Lee *et al.* 2021). This is associated with a gradual reduction in strength (Chang *et al.* 2017). Concerns regarding the moisture-dependent strength performance and durability of biopolymer-treated soils hinder their field applicability, and chemical crosslinking is considered a viable solution to the resolution of this problem.

Crosslinking involves joining two or more polymers via covalent or noncovalent connections to form extended and 3-dimensional polymer networks. Crosslinking methods to improve polysaccharide biopolymer-based materials have been widely used for biomedical (Oh *et al.* 2009, Reddy *et al.* 2015), film/coating (Lan *et al.* 2010), and environmental applications (Tungittiplakorn *et al.* 2004). Crosslinking of polysaccharide biopolymers reduces the mobility of the polymer structure and usually enhances its mechanical properties and water resistance, reducing its water solubility and swelling capacity. For geotechnical engineering purposes, Im *et al.* (2020) assessed the feasibility of malonic acid-based crosslinking to reduce the water reactivity of starch biopolymer-treated soils (Im *et al.* 2021). Although the crosslinked starch biopolymer enhances the soil's wet strength by reducing the available hydroxyl groups and increasing polymer connections, in situ mixing with soil is challenging as the reaction process of crosslinking necessitates activation heat. Hence, it is necessary to study straightforward crosslinking strategies that combine feasibility and affordability in advanced field applications for biopolymer-soil treatment

Without the use of heat, some divalent salts (e.g.,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Ba}^{2+}$ ) can cause the gelation or precipitation of polysaccharide biopolymers under alkaline environment (Shibaev *et al.* 2020). Particularly  $\text{Ca}^{2+}$  is favored over other monovalent and divalent cations because of its affinity and superior gel strength (Sutherland 1994). Meanwhile,  $\text{Na}^+$  is one of the most popular retarders for controlling gelation time in polymer crosslinking through the charge screening effect (Koochi *et al.* 2011).

Therefore, in this study, we explored calcium- and sodium-ion-induced crosslinking methods under alkaline conditions to promote the gelation of polysaccharide biopolymers and enhance wet strength and durability of biopolymer-treated soil. In addition, based on the laboratory test results, we conducted a field-scale implementation of a crosslinked biopolymer-treated soil mixture via in situ compaction and pressurized spraying for slope surface protection.

## 2. Materials and methods

### 2.1 Used soil

In this study, site soil and Red yellow soil were utilized to investigate the strengthening effect of biopolymer treatment in the laboratory. The soil was obtained from a construction site in Musu-dong (36°27'60" N, 127°40'04")

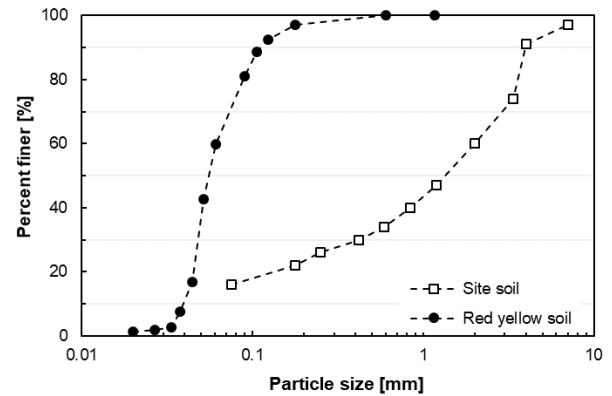


Fig. 1 Particle size distribution of the used soil

Table 1 Soil properties of the used soil

Used soil	Mean particle size [mm]	Specific gravity	Fine fraction [%]	USCS*
Site soil	1.2	2.67	16	SM (silty sand)
Red Yellow soil	0.058	2.70	70	CL (lean clay)

\*Unified Soil Classification System

in Daejeon, South Korea. Fig. 1 shows the particle size distribution of the Musu-dong site soil and Red yellow soil obtained by a sieve analysis (ASTM 2017). The site soil was classified as silty sand (SM) with a mean particle size ( $D_{50}$ ) of 1.2 mm, a specific gravity ( $G_s$ ) of 2.67, and a fine fraction of 16%. Red yellow soil (also called Hwangtoh), classified as lean clay (CL), has a  $D_{50}$  of 0.058 mm,  $G_s$  of 2.7, and a fine fraction of 70%. Both soils were dried in an oven at 110°C for 24 h before being mixed with the biopolymer.

### 2.2 Biopolymer: xanthan gum-starch compound

This study mainly focused on the xanthan gum (XG) and corn starch (ST) compound biopolymer, composed of a 3:7 mixture of XG and ST that considers the soil strengthening effect and economic feasibility (Seo *et al.* 2021). The XG-ST (3:7) compound biopolymer (XS) has shown satisfactory performance in previous studies as a slope surface strengthening material for levee protection to mitigate surface soil erosion (Ko and Kang 2018; Tran *et al.* 2019; Kang *et al.* 2021). The details of the individual materials are described below.

XG, produced by *Xanthomonas campestris*, is a common and successfully commercialized biopolymer. The XG molecule has a trisaccharide chain including carboxyl groups (-COOH), which provide an anionic charge on the molecular structure. Thus, XG can efficiently adsorb water molecules and form viscous hydrogels via hydrogen bonding between trisaccharide side chains aligned with glucose (García-Ochoa *et al.* 2000). XG has been widely utilized in the food, cosmetic, pharmaceutical, and oil industries because it effectively increases the viscosity of

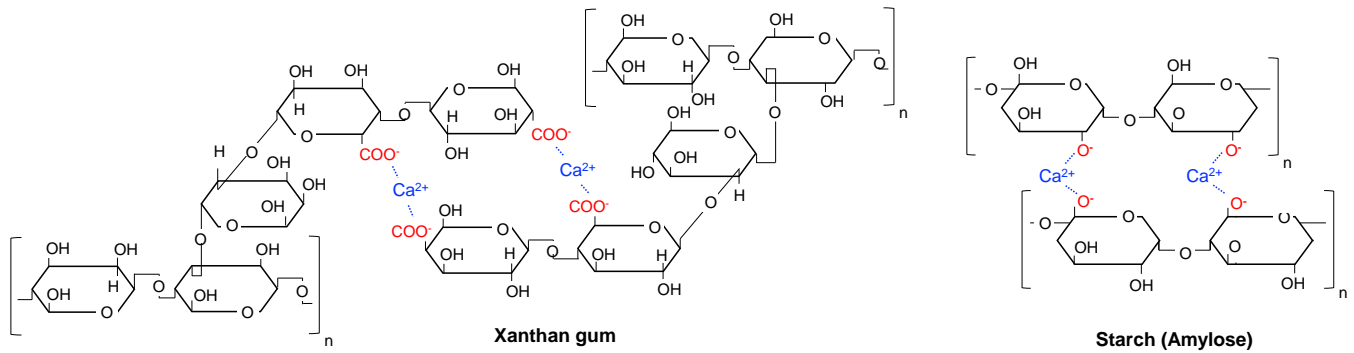


Fig. 2 Proposed mechanism of crosslink in xanthan gum biopolymer and starch (amylose)

Table 2 Overall biopolymer and crosslinker mixing recipes for gel and soil sample preparation

Specimen	Soil [g]	Deionized Water [g]	Biopolymer		Ca(OH) <sub>2</sub> [g]	NaOH [g]
			Xanthan gum [g]	Starch [g]		
CaXS Gel (Gel A, B, C, D, E)	-	250	4.5	10.5	0.5, 1, 1.5, 2.5, 5	-
CaNaXS Gel (Gel F, G, H, I)	-	250	4.5	10.5	5	0.5, 1, 1.5, 2.5, 5
	1000	250	4.5	10.5	5	0, 2.5, 5, 10
CaNaXS-soil (Site soil)	1000	250	4.5	10.5	5	0.5
	1000	250	3.0	7	5	0.5
	1000	250	1.5	3.3	5	0.5
CaNaXS-soil (Red yellow soil)	1000	250	4.5	10.5	5	5

the solvent, even in small amounts with stability to temperature and pH variations (Sworn 2021). Currently, XG is used as a soil-strengthening agent in geotechnical engineering (Singh and Das 2020).

ST, which is composed of two polysaccharides (i.e., amylose and amylopectin), is one of the most commercialized biopolymers in many industries. Various ST subtypes exist based on the plants from which they originate (e.g., corn, potato, and casaba). The ratio of amylose to amylopectin and the molecular length of ST vary considerably depending on the source, and different ratios of the components affect the rheological and chemical properties of ST (Perritano 2018).

This study used corn ST, which contains approximately 25% amylose. Amylose can induce gelation when ST is dissolved in water heated above 90°C, resulting in a firm gel when cooled, whereas amylopectin forms swollen granules that increase fluid viscosity (Eliasson 2017). Combinations of ST and other biopolymers have been investigated for use as soil-binding materials owing to their affordability (Ayeldeen *et al.* 2016, Kulshreshtha *et al.* 2017, Im *et al.* 2021).

### 2.3 Reactants for cation crosslinking

Previous studies have documented that inorganic polyvalent salts, including Ca<sup>2+</sup>, Mg<sup>2+</sup>, Ba<sup>2+</sup>, Fe<sup>3+</sup>, and Cr<sup>3+</sup>, can cause the gelation or precipitation of polysaccharide biopolymers (Shibaev *et al.* 2020, Wang *et al.* 2022). The area of reactivity is specific to the molecular mass of the

cation and the polymer concentration involved; hence, the type of anion seems to have little or no influence (Bergmann *et al.* 2008). Among divalent ions that require a high pH for gelation (i.e., alkaline gelatinization), Ca<sup>2+</sup> is strongly associated with the molecular strands of the polymer chain owing to its high affinity for ionic attraction and because the geometry of the long-chain polymer-calcium complex is favored over other mono- and divalent cations (Sutherland 1994). Although trivalent salts induce the polysaccharide biopolymer gelation in a wide pH range, they are limited owing to their high cost compared to mono- and divalent salt. Hence, calcium hydroxide (Ca(OH)<sub>2</sub>, CAS No. 1305-62-0) was utilized as an inexpensive and straightforward cation crosslinker to enhance the XS biopolymer for cation crosslinking-induced gelation of XS in this study, considering the affordability in geotechnical engineering practices.

Ca(OH)<sub>2</sub> is generally utilized as a component of lime mortar in civil construction areas and as a lime-based fertilizer in agricultural soil engineering (i.e., antimicrobial activity and supply calcium to vegetation) (Matsuzaki *et al.* 2021). It has been demonstrated that Ca(OH)<sub>2</sub> solution induces gelatinization of XG and ST biopolymer solutions by forming complex inter-/intra-molecular connections between biopolymer chains and cations (Maher 1983, Harada *et al.* 1991, Bryant and Hamaker 1997, Patel *et al.* 2020). Under strong basic conditions, Ca<sup>2+</sup> interacts with deprotonated hydroxyl (-OH) and carboxyl (-COOH) groups in polysaccharide biopolymer chains by physical crosslinking via van der Waals interactions (Cornejo-

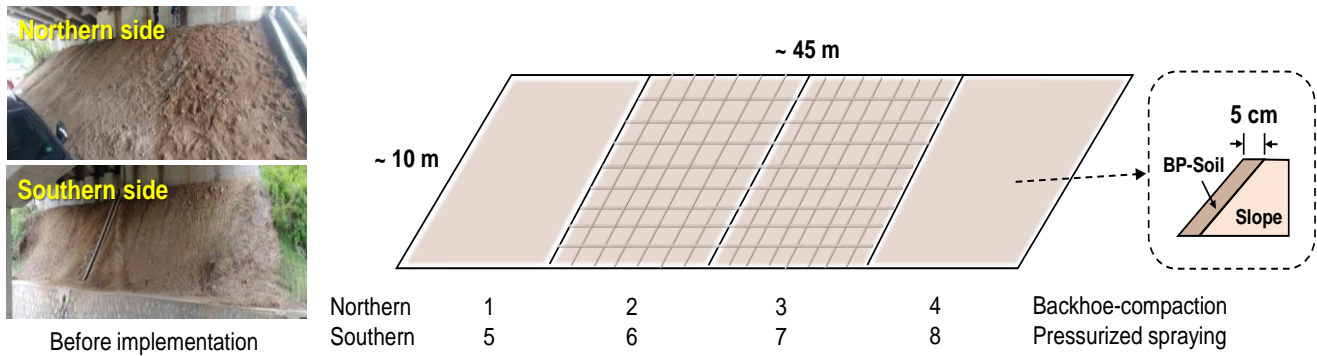


Fig. 3 Schematic illustration of site slope with panorama view of the site (before implementation)

Table 3 Detailed field implementation conditions for each slope section

Location	Northern side				Southern side			
Section	1	2	3	4	5	6	7	8
Material	Ca(OH) <sub>2</sub> -NaOH crosslinked XS compounds mixed with site soil							
Method	Spreading and compaction using the backhoe				Pressurized-spraying			
BP-soil installation thickness [cm]	5	5	5	5	5	5	5	5
Installation of steel mesh	None	Done	Done	None	None	Done	Done	None
Construction area [m <sup>2</sup> ]	100	125	125	100	100	125	125	100
BP-soil volume [m <sup>3</sup> ]	5	6.25	6.25	5	5	6.25	6.25	5
Additional watering	-	-	-	-	None	None	Done	Done

Villegas *et al.* 2018, Boonkanon *et al.* 2021), as shown in Fig. 2.

Sodium hydroxide (NaOH, Cas No. 1310-73-2), also called caustic soda, is used as an additional retarder for crosslinking between divalent cations and polymer chains. NaOH, the most widely used base in chemical experiments, is commonly used in the soap, paper, pulp, and food industries because of its reactivity and economic efficiency (Park *et al.* 2017, Hassan *et al.* 2018). NaOH has been utilized as a crosslinking retarder to control gelation time by providing repulsion between ions and carbonic groups on chains of the co-polymer (i.e., the charge screening effect) (Koochi *et al.* 2011). Ca(OH)<sub>2</sub> and NaOH in analytical grade (Daejung Chemical Co., Korea) with a purity of > 99% were used as sources of Ca<sup>2+</sup> and Na<sup>+</sup> in this study.

#### 2.4 Preparation of crosslinked biopolymer gel and biopolymer-treated soil specimen

Crosslinked biopolymer-soil specimens were produced in two steps: 1) preparation of crosslinked gel by the dissolution of XS, Ca(OH)<sub>2</sub>, and NaOH in deionized water and 2) mixing with soil and molding. The overall mixing compositions of the biopolymer and crosslinkers evaluated in this study are summarized in Table 2. Only Ca(OH)<sub>2</sub> crosslinked XS (i.e., CaXS) gel and both Ca(OH)<sub>2</sub>-NaOH crosslinked XS (i.e., CaNaXS) gels were prepared through step 1 to determine the gel formation properties and approximate gelling time. Among these gels, feasible candidate recipes were selected, and CaNaXS-treated soil specimens were prepared with different retarder (NaOH)

contents. After that, the final candidate recipe-treated soil samples were prepared with different biopolymer contents (i.e.,  $m_{xs}/m_s$  = ratio of XS mass to soil mass) and soil types with different fine contents (i.e., site soil and Red Yellow soil) for comparison.

The detailed process of the gel and gel-treated soil samples is as follows. First, the XS biopolymer and crosslinkers were thoroughly mixed in a powdered state according to the mixing recipe. The powdery-state compounds were dissolved in deionized water using a laboratory hand mixer at high speed (20,000 rpm) for 30 s to achieve uniform mixing. After preparing the crosslinked gel, it was uniformly blended with the target soil, resulting in  $m_{xs}/m_s$  of 0.5, 1, and 1.5%, whereas equal initial water content ( $m_w/m_s$ ) of a mixture of 25%.

#### 2.5 Unconfined compressive test

Unconfined compressive strength (UCS) tests were performed to assess the strength of crosslinked XS-treated soil specimens molded in a 50 mm × 50 mm × 50 mm cubic shape. The UCS tests were conducted using a master loader (HM-5030.3F) at a constant axial loading rate of 0.4 mm/min (1% per minute), according to ASTM D1633. Three samples were measured to obtain the average UCS values for each condition (ASTM 2017). In this study, the strength of the CaNaXS-treated soil was assessed in terms of two moisture states: wet and dry. The wet strength indicates that the UCS of the soil sample was cured for two weeks without evaporation, whereas the dry strength indicates that the soil sample was dried at room temperature



Fig. 4 Field implementation procedure of crosslinked biopolymer-treated soil

(23°C) until the weight remained constant.

Additionally, CaNaXS-treated sand samples were subjected to repeated wetting and drying cycles with UCS measurements at each interval according to ASTM D559 to investigate durability under cyclic wetting–drying conditions (ASTM 2015). The cycle comprised air dehydration for 48 h (drying phase) and re-saturating samples in tap water for 24 h (wetting phase). Four wetting and drying cycles were conducted.

## 2.6 Field implementations

### 2.6.1 Site of interest and implementation conditions

Field implementation of the CaNaXS-treated site soil was conducted from July 25<sup>th</sup> to July 26<sup>th</sup>, 2021, on steep slopes near the bridge abutment of the highway (Daejeon, Korea; 36°16'31"N, 127°24'12"E), as shown in Fig. 3. During the site implementation period, the daily temperature of the site varied between 24°C and 35°C, without precipitation. The CaNaXS-treated site soil was implemented on eight trapezoid sections (four in the northern part and four in the southern part) with a surface area of approximately 100–125 m<sup>2</sup> and a slope angle of 35–40° (Table 3). The detailed mixing recipes for the CaNaXS-treated soils are described in Table 4. The composition of the biopolymer-soil mixing was determined based on the results of the laboratory experiments described in Chapter 3. According to Seo *et al.* (2021), the designed final water content was determined to be 27–29%, considering the sum of the water contained in the prepared CaNaXS solution (i.e., the amount of water is approximately 20% of the soil mass) and 7–9% of the natural water content already included in the site soil.

### 2.6.2 Site implementation procedure

The CaNaXS-treated site soil was installed in two ways: 1) in-situ spreading and compaction method using a backhoe and 2) pressurized spraying method using a soil-spraying machine. Fig. 4 illustrates the overall procedure for the preparation and implementation of CaNaXS-treated site soil.

The biopolymers and crosslinkers, both in the powder phase, were thoroughly hydrated with water using an in-situ mixer drill (Fig. 4(a)). The crosslinked biopolymer solution was then poured into a large box (i.e., a volume of 2 m<sup>3</sup>) and mixed with the site soil to a volume of 1 m<sup>3</sup> for approximately 20 min (Fig. 4(b)) according to Seo *et al.* (2021). Simultaneously, the natural surface soil on the slope was cleaned by plowing and flattening using a backhoe. A steel wire mesh in a square grid pattern was then installed in Sections 2, 3, 6, and 7 to verify whether it prevented the unset CaNaXS-treated site soil from flowing down the slope (Fig.4c). Subsequently, the CaNaXS-treated site soil was sequentially installed on each slope section. In sections 1 to 4, the CaNaXS-treated site soil was first spread on a slope, flattened by workers (Fig. 4(d)), and then compacted using a backhoe (Fig. 4(e)). Meanwhile, in sections 5 to 8, the CaNaXS-treated site soil was introduced into the spraying machine, which shoots the soil mixtures at the end of the nozzle using pneumatic pressure (Figs. 4(f) and 4(g)). Seo *et al.* (2021) documented that field-scale spraying of biopolymer-treated soil requires a higher water mixing ratio to ensure adequate pumping and adhesion of the sprayed mixture. Thus, in Sections 7 and 8, watering systems were attached to the end of the nozzle, and extra water was added during the spraying process to investigate the effect of watering on spraying efficiency (Seo *et al.* 2021). CaNaXS-



Fig. 5 Panorama view of the site after implementation

Table 4 Detailed composition of CaNaXS-treated soil implemented in the field

Detail Composition of binder and crosslinker [% to soil mass]					Water content [%]	
Overall biopolymer <sup>a)</sup>	Xanthan gum	Starch	Ca(OH) <sub>2</sub>	NaOH	Site soil <sup>b)</sup>	CaNaXS-Soil <sup>c)</sup>
1.50	0.45	1.05	0.5	0.5	7	27-29

<sup>a)</sup> Total biopolymer content (Xanthan gum + Starch) to the mass of soil

<sup>b)</sup> Natural water content to soil ratio in mass (%) of site soil

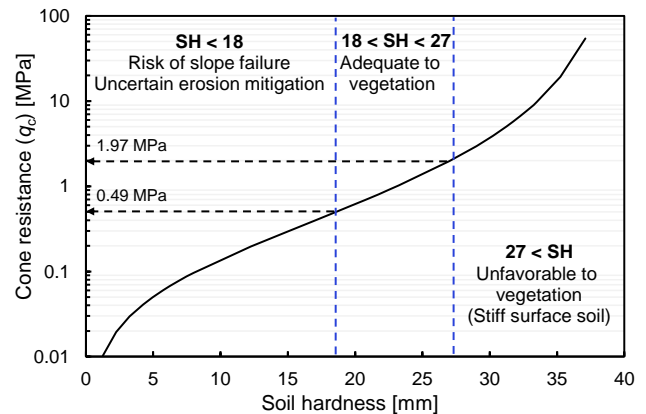
<sup>c)</sup> Water content to soil ratio in mass for site soil + biopolymer + water mixing

treated soil was implemented to a thickness of 5 cm for all sections on the slope using two methods (Fig. 2 and Table 3), and the panoramas of the slope sprayed with the BP-soil mixture immediately after implementation are shown in Fig. 5.

### 2.7 Field monitoring: surface soil hardness measurement

Field monitoring was conducted five times for approximately one year to analyze the post-strength performance of the implemented biopolymer-treated soil layer. According to the design criteria for slope construction in South Korea (MOLIT 2009), construction and maintenance standards present soil hardness as the primary factor when reinforcing the slope surface layer through soil-seed spraying. A cone-type portable penetrometer (i.e., Yamanaka-type penetrometer) was used to assess the soil hardness of the biopolymer-treated surface layer. Yamanaka-type penetrometers have been widely used for the in situ assessment of soil strength for slope failure stability analysis, sediment cores, and convenient strength evaluation of engineered soils (Wakatsuki *et al.* 2005, Wu *et al.* 2011, Shimizu and Ono 2016, Wang and Chen 2017).

It is composed of a metal cone with a cone top angle of 30° and a cone cross-sectional area of 2 cm<sup>2</sup>, with a spring (i.e., spring constant = 2 kg/cm) (Yamanaka and Matsuo 1962). The cone was pressed perpendicularly to the soil surface, and the backward distance until the cone stopped penetrating was measured, which was defined as the soil hardness ( $w$ , in mm). The cone resistance ( $q_c$ , in kPa) was calculated by dividing the weight corresponding to the spring contraction by the cross-sectional area of the cone in contact with the soil. The equation to convert the measured soil hardness to  $q_c$  of the soil is as follows (NIAST 2000)

Fig. 6 Relationship between cone resistance ( $q_c$ ) and soil hardness

$$q_c \text{ [kPa]} = \frac{10w}{0.811 \times (40-w)^2} \quad (1)$$

According to the soil hardness criteria in Road slope revegetation method: design and construction guideline from the Ministry of Land Infrastructure and Transport (MOLIT) of South Korea, risk monitoring for slope failure and assessment of erosion resistance of surface soils are recommended when the soil hardness is less than 18 mm. Soil hardness in the range 18–27 mm is adequate for vegetation growth without the risk of slope failure, whereas soil hardness over 27 mm indicates very high stiffness in surface soil that is disadvantageous for plant growth and root invasion (Fig. 6) (MOLIT 2009; Chen *et al.* 2013). In this study, the surface hardness of the CaNaXS-treated soil on the slope was measured 10 times and averaged for each section.



Fig. 7 CaNaXS Gel uniformity with various crosslinker ratios

Table 5 Visual observation results on gel formation behavior

Recipe	XS compounds [% to soil]	Ca(OH) <sub>2</sub> [% to soil]	NaOH [% to soil]	Approximate gelling time (hardening period) [min]	Gel texture description
Gel A	1.5	0.05	-	N/A	No gelling occurred
Gel B	1.5	0.1	-	N/A	No gelling occurred
Gel C	1.5	0.15	-	60 (120)	Soft and uniform gel
Gel D	1.5	0.25	-	15 (60)	Stiff gel with moderate uniformity
Gel E	1.5	0.5	-	< 5 (30)	The very stiff gel formed in a short time showing poor uniformity with the fast formation of gel aggregates
Gel F	1.5	0.5	0.05	< 5 (30)	Very stiff gel with a rough surface and poor uniformity
Gel G	1.5	0.5	0.10	6-7 (45)	Very stiff gel with a rough surface and poor uniformity
Gel H	1.5	0.5	0.25	15 (60)	Stiff gel with moderate uniformity
Gel I	1.5	0.5	0.5	30 (75)	Stiff and uniform gel

### 3. Experimental results and analysis

#### 3.1 Visual observation of crosslinking-induced gel formation behavior

Gel formation characteristics, including the approximate gelling time and appearance of a total of nine recipes (i.e., five CaXS gels and four CaNaXS gels), were visually inspected to select candidate recipes for further biopolymer-soil mixing processes (Table 5). The crosslinked gel was thoroughly mixed for 30 s and then gelled in a 6 cm diameter aluminum dish (Fig. 7).

CaXS gels (Gel A to E) prepared by mixing deionized water with XS and Ca(OH)<sub>2</sub> at  $m_{ca}/m_{xs} = 3, 6, 10, 16,$  and  $33\%$  (i.e.,  $m_{ca}/m_{xs} =$  ratio of Ca(OH)<sub>2</sub> mass to XS mass) as shown in Table 2. The results showed that the initial hardening of the gel was observed in 5-15 minutes and the stiffness gradually increased over time for approximately 30-60 minutes when 16% and 33%  $m_{ca}/m_{xs}$  of Ca(OH)<sub>2</sub> was added to the XS hydrogel. When 10%  $m_{ca}/m_{xs}$  of Ca(OH)<sub>2</sub> was added, initial gelation took approximately 60 min, but the gel stiffness was much softer than that of Gel A and B, and no additional hardening behavior was observed. On the other hand, gelation was hardly observed when  $m_{ca}/m_{xs}$  less than 6% was added, indicating that viscous XS hydrogels can build stiff gelling structures above a certain crosslinker content compared to polymer contents (Reddy *et al.* 2015; GhavamiNejad *et al.* 2020). Moreover, the more Ca(OH)<sub>2</sub> is crosslinked to XS, the faster gelation occurs with high stiffness; however, the speed of gelation induces variation in gel uniformity (Kuo and Ma 2001). For example, in Gel E, irregular aggregates formed immediately after mixing because of the frequent reaction between XS and calcium ions; as a result, the gel uniformity was very poor (Fig. 7).

In response, we conclude that the crosslinking reaction speed of the XS-Ca<sup>2+</sup> network must be controlled or slowed by the addition of a retarder (NaOH) to achieve more uniform gels and homogeneous mixing with soil.

To delay the reaction time, CaNaXS gels (Gel F to I) were prepared by mixing deionized water with XS-Ca(OH)<sub>2</sub> ( $m_{ca}/m_{xs} = 33\%$ ) and NaOH with 10, 20, 50, and 100% Ca(OH)<sub>2</sub>. The results showed that adding NaOH increased the approximate gelling time of CaNaXS gels with a decrease in stiffness because the monovalent ion allows much looser condensation than when the negative charges of the polymer chain are shielded by divalent ions (Sircar *et al.* 2013). On the other hand, fast precipitation still occurs in Gels F and G, where the mass ratio of NaOH to Ca(OH)<sub>2</sub> is less than 50%, exhibiting locally formed micro-aggregates and poor uniformity with rough surfaces (Fig. 7). Gel I, composed of a 1:1 ratio of Ca(OH)<sub>2</sub> and NaOH, was selected as a candidate recipe given the time required to mix and implement in the field and the gel uniformity, which affects the stability of the soil after mixing.

#### 3.2 Unconfined compressive strength of crosslinked biopolymer-treated soil

The unconfined compressive strength (UCS) of crosslinked biopolymer-treated soil is essential for evaluating the soil stabilizing performance. The UCS of crosslinked biopolymer (CaNaXS)-treated soil samples was assessed with various crosslinker compositions, biopolymer concentrations, soil types, and strength responses during the wetting-drying process (Fig. 8). Fig. 8(a) depicts the UCS of biopolymer-treated site soil with different Ca(OH)<sub>2</sub> and NaOH ratios. When only Ca(OH)<sub>2</sub> was crosslinked to XS, the rapid reaction time caused gel disturbances during

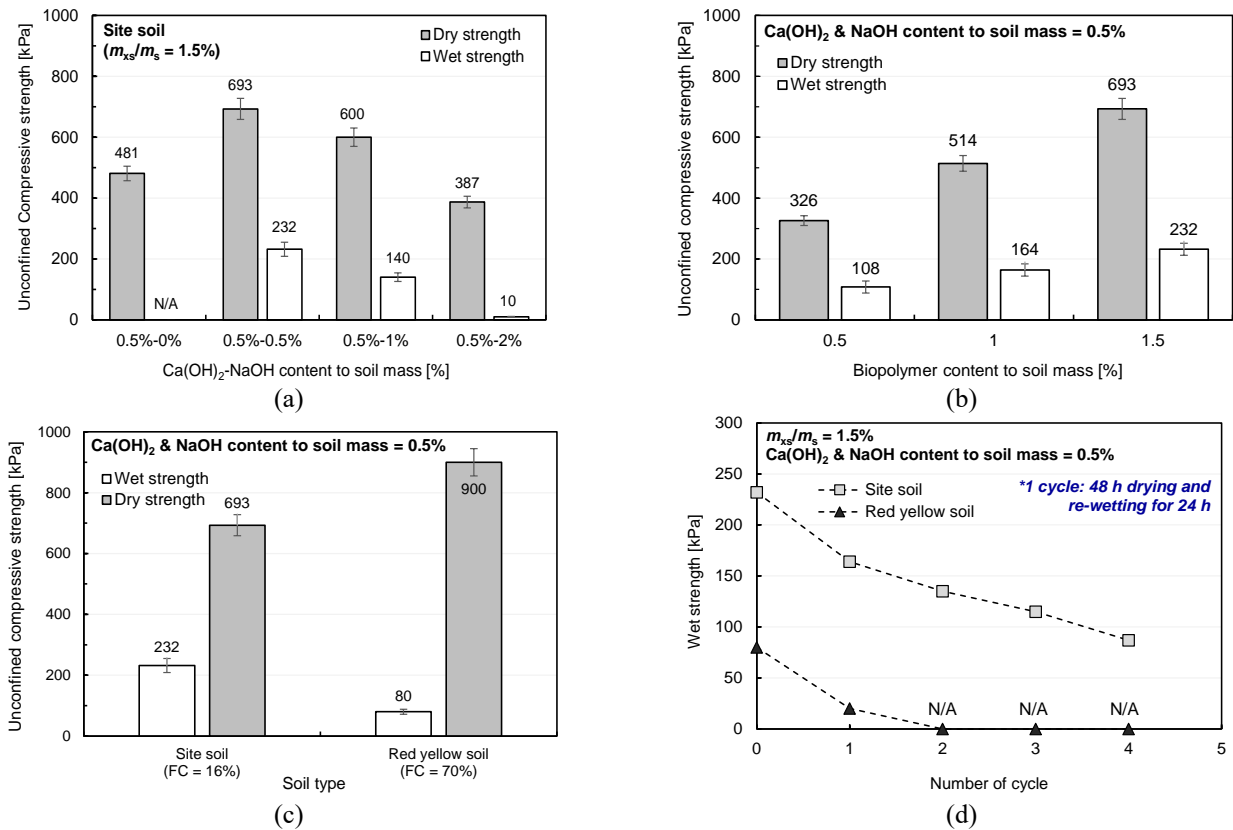


Fig. 8 Unconfined compressive strength of crosslinked biopolymer-treated soil

mixing with the soil, resulting in negligible wet strength. Meanwhile, gelation was retarded when NaOH was added, allowing it to be thoroughly mixed with the soil, and the wet strength was approximately 230 kPa. When more NaOH was added, the wet strength gradually decreased, which was consistent with the observations of the gel formation test (Table 5). In addition, both dry and wet strengths increased as the biopolymer concentration increased (Fig. 8(b)). Furthermore, it was noted that the dry UCS increased by 30%, whereas the wet UCS decreased by 65% when the mixed soil had a larger fine fraction (Fig. 8(c)). This implies that the larger fine fraction interfered with crosslinking by competitive cation adsorption between the clay surface and the polymer chain (Garver *et al.* 1989). Thus, it is presumed that the clay particles interrupted the intermolecular connection between XS and cations, resulting in a less significant strengthening effect in the clay-rich soil. Fig. 8(d) presents the wet UCS after repeated drying and rewetting of the CaNaXS-treated soils. Although both types of soil experienced a considerable strength reduction after the first cycle, the CaNaXS-treated site soil samples showed a residual wet UCS of 30% after four cycles, whereas the Red yellow soil samples lost all strength due to severe swelling after the 2<sup>nd</sup> cycle. It is believed that not only was the strength due to gel stiffness not facilitated because the crosslinking was interfered with by the clay particles but the degradation was also accelerated because of the synergetic water absorption of the residual biopolymer and the clay particles that did not bind (Lee *et al.* 2022).

#### 4. Field implementation monitoring results and analysis

After implementing the CaNaXS-treated soil on the slope, as described in Section 2.6.2, the soil hardness of the slope surface was monitored periodically for one year. Monthly variations in temperature and precipitation at a construction site and the monitoring results of each slope section are presented in Figs. 9 and 10, respectively.

The monitoring data showed that soil hardness and consequent cone resistance ( $q_c$ ) of CaNaXS-treated slope were 1.7 - 2 times and 5 - 10 times of average SH and  $q_c$  value of untreated natural slope (control) satisfying the criteria on risk for slope failure and erosion (i.e., the blue line in Fig. 10(a)), even though soil hardness and  $q_c$  have slightly fluctuated following seasonal climate variation. Furthermore, higher soil hardness and  $q_c$  were observed when CaNaXS-treated soil was implemented by the pressurized spraying method (i.e., Sections 5 to 8). This suggests that the pressurized spraying method has the potential to acquire high density (i.e., strength) as well as construction efficiency, despite a shorter construction time (40 min per section) compared to the in situ compaction method (1.5 h per section).

Conversely, the edge regions of sections 1, 4, 5, and 8, which are more severely exposed to climatic weathering conditions, were divided into the outer, boundary, and inner zones, showing distinct responses (Fig. 11). In detail, soil hardness less than or around the stability criteria (18 mm) was observed in the outer zone of each section (15-50%

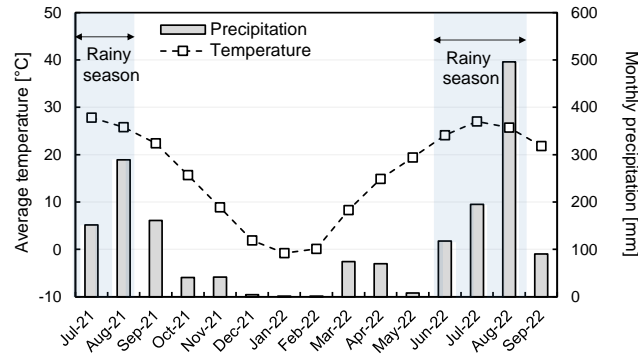


Fig. 9 Average monthly temperatures and precipitations in South Korea (Daejeon) from July 2021 to September 2022. Data from Korea Meteorological Administration ([https:// www. weather. go.kr](https://www.weather.go.kr))

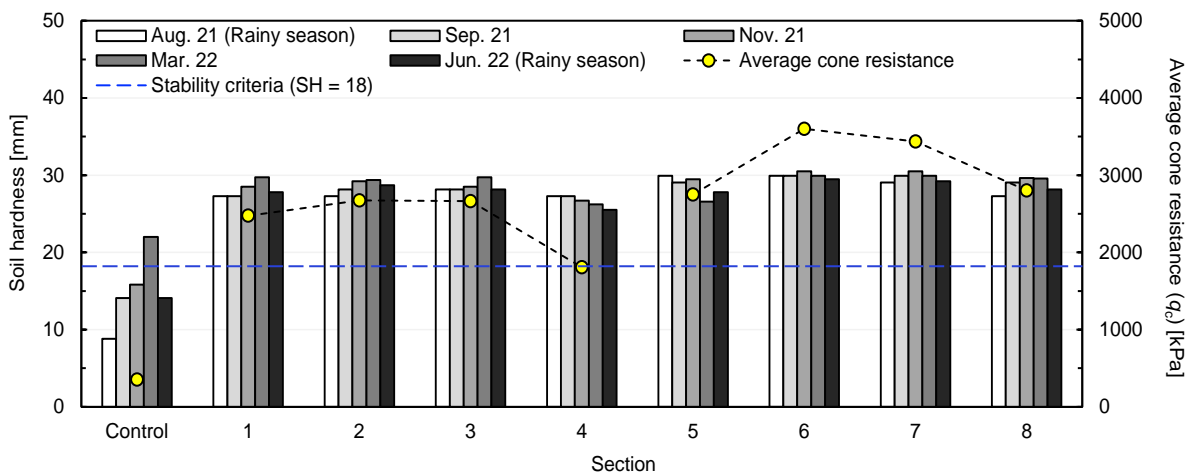


Fig. 10 Soil hardness and average cone resistance of all sections

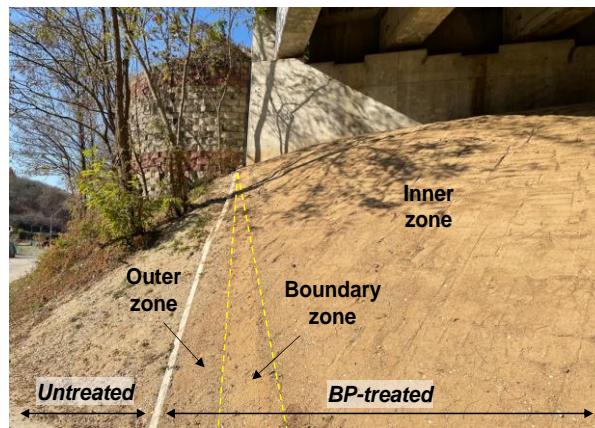


Fig. 11 Untreated region, outer, boundary, and inner zones in section 1

reduction), and  $q_c$  values decreased to 71 – 88% of those of the inner zone (Fig. 12) regardless of the implementation method. As we can see from the durability test results (Fig. 8d), the overall strength of the CaNaXS-treated soil on the slope deteriorated for a year owing to the climatic weathering effect, including repeated dehydration and re-wetting processes. Although a portion of the surface soil (i.e., approximately 2 cm out of 10 cm thickness) was

degraded with no bulk failure or detachment of the stabilized layer, these findings suggest that there are potential concerns about the long-term durability of CaNaXS-treated soil. There is a possibility of physical disturbance (i.e., gel fracturing) of hardened gel during soil mixing and implementation due to the short gelling time within a few hours and lower homogeneity in field-mixed biopolymer-treated soil compared to indoor-mixed soil.

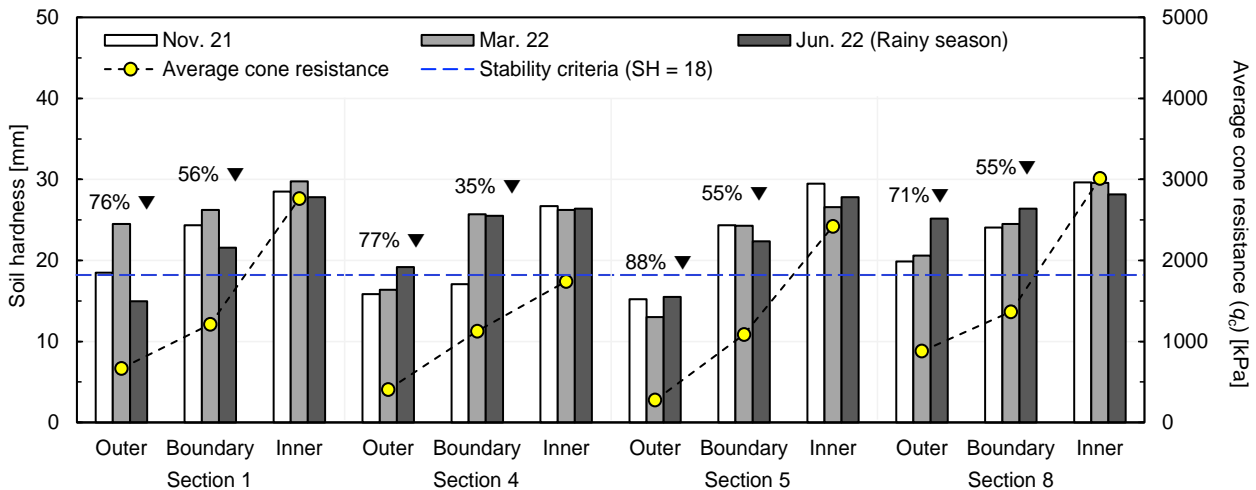


Fig. 12 Soil hardness and average cone resistance of three regions in section 1, 4, 5 and 8

In summary, it was confirmed that a stiff gel was formed through alkaline-based cation crosslinking of the XS biopolymer and feasibility in field implementation using the existing method. However, it is difficult to control gelation finely in the field construction process, which is regarded as a vital factor for long-term stability. Thus, recipe improvement using trivalent cation crosslinking, which has a longer gelation period (i.e., a few days), is recommended to attempt in further study.

## 5. Conclusions

This study explored the feasibility of alkaline gelatinization in xanthan gum-starch biopolymers (XS) and its field applicability as a slope protection material. The indoor experimental results demonstrated that the XS hydrogel could form a stiff gel under alkaline conditions with  $\text{Ca}^{2+}$ , and the gelation speed and consequent gel stiffness and uniformity could be varied by the concentration of  $\text{Ca}^{2+}$  and the presence of monovalent ions as retarders. Furthermore, stiff gel formation by Ca-Na crosslinking in the XS hydrogel increased the wet strength of the XS-treated soil. As an advanced slope-protection material, CaNaXS-treated soil was implemented on a field slope and monitored for a year after construction. We confirmed that the surface soil hardness was significantly increased; in particular, the pressurized spraying method was adequate compared to the compaction method considering the higher strength (i.e., soil hardness and cone index) and temporal construction efficiency.

These results demonstrate the feasibility of alkaline-based cation crosslinking as an advanced strategy for biopolymer-soil treatment techniques. However, because gradual degradation by the climatic weathering effect still occurred, we suggest the necessity of further recipe improvement by using trivalent cations, which are known to have more extended gelling periods.

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