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Enhancing the wet resistance of biopolymer-treated sands by crosslinking agar and xanthan gum

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ABSTRACT

Geotechnical construction often presents considerable environmental challenges, leading to growing interest in sustainable alternatives such as biopolymers. Among these, polysaccharide-based biopolymers have gained attention, owing to their natural abundance and ability to improve soil properties. However, their inherent hydrophilic nature can cause strength degradation upon repeated moisture exposures, limiting their long-term durability. To address these limitations, this study investigates the effectiveness of cross-linked polysaccharide biopolymers—specifically, Xanthan gum and Agar gum. Uniaxial compressive strength (UCS) tests were conducted on Jumunjin sand specimens treated with varying Xanthan gum-to-Agar mass ratios (i.e., 0%, 25%, 50%, 75% and 100%) and total biopolymer-to-soil mass ratios (i.e., 0.5% and 2%). Experimental variables included drying duration (i.e., 1, 3 and 7 days), and submergence conditions to assess water resistance and recovery. The results revealed that biopolymer cross-linking significantly improved UCS and water resistance. Notably, specimens with moderate Agar gum-to-Xanthan gum ratios exhibited enhanced durability under wet conditions. These findings demonstrate the potential of AG-XG cross-linking as an environmentally friendly grouting method for tunnel and underground space construction in sandy soils.

1. INTRODUCTION

Granular materials like sand typically require cementation to enhance their mechanical properties (Wu et al., 2018), especially for constructing large-scale infrastructures such as tunnels, airports, and underground spaces (He et al., 2020).

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Cement and chemical materials, such as polyurethane and polyacrylate, have been widely used to cement sand particles through pore penetration (Abraham et al., 2013; Mollamahmutoğlu et al., 2017). However, the production of these materials poses significant environmental concerns. For example, manufacturing one ton of Portland cement clinker releases approximately 0.866 tons of greenhouse gases (Barcelo et al., 2013). Furthermore, cement elevates soil pH from around 5.6 to highly alkaline levels (e.g., pH = 9), due to the hydrolysis of calcium hydroxide and magnesium oxide (Richman et al., 2006). Chemical grouting also presents health risks; polyurethane can cause irritation and allergic reactions, while polyacrylate is known as an irritant (Vik et al., 2000). Furthermore, chemical grouts can be leached into groundwater aquifers, posing additional environmental threats (Bonacci et al., 2009; Vik et al., 2000).

Biopolymers, derived from natural sources such as plants and microorganisms, have attracted attention as sustainable soil stabilization agents due to their minimal environmental impacts (Cole et al., 2012). However, each biopolymer type exhibits specific limitations, such as low wet resistance due to their hydrophilicity or insufficient strength development (Kumar et al., 2024). To address these limitations, this study investigates the potential of crosslinking thermogelling agar with hydrophilic xanthan gum (Kumar et al., 2024). Specifically, the unconfined compressive strength of sand treated with various agar-to-xanthan gum ratios was analyzed over different drying periods. The results highlight the potential of agar to enhance soil's durability under submerged conditions.

2. MATERIALS AND METHODS

2.1 Materials

2.1.1 Agar (AG)

Agar ($(C_{12}H_{18}O_9)_n$) is a polysaccharide biopolymer derived from red algae (*Rhodophyceae*). It exhibits thermo-gelation properties through hydrogen bonding with water, melting at temperatures between 85 and 95 °C and gelling between 32 and 45 °C (Jang et al., 2024). AG gel demonstrates water resistance due to the formation of helical dimers (N. Rhein-Knudsen et al., 2015). In this study, powdered AG (CAS: 9002-18-0; Sigma Aldrich Co., Portugal) was used without any purification. AG has a light yellow to beige color, and its 1.5% aqueous solution exhibits a pH ranging from 6.0 to 8.0 (Sigma-Aldrich, 2025a).

2.1.2 Xanthan gum (XG)

Xanthan gum ($(C_{35}H_{49}O_{29})_n$) is a polysaccharide biopolymer produced by the metabolism of the bacterium *Xanthomonas Campestris*. It forms a viscous hydrogel through hydrogen bonding with water and is characterized by its hydrophilic properties. Previous studies have indicated that XG shows relatively higher improvement in soil strength compared to AG; however, due to its hydrophilic nature, XG lacks water resistance (Kumar et al., 2024; Lee et al., 2022). The powdered XG used in this study (CAS: 11138-66-2; Sigma Aldrich Co., US) has a faint yellow to beige coloration, and

exhibits a Brookfield viscosity of 800 – 1200 cps in a 1% aqueous solution (Sigma-Aldrich, 2025b).

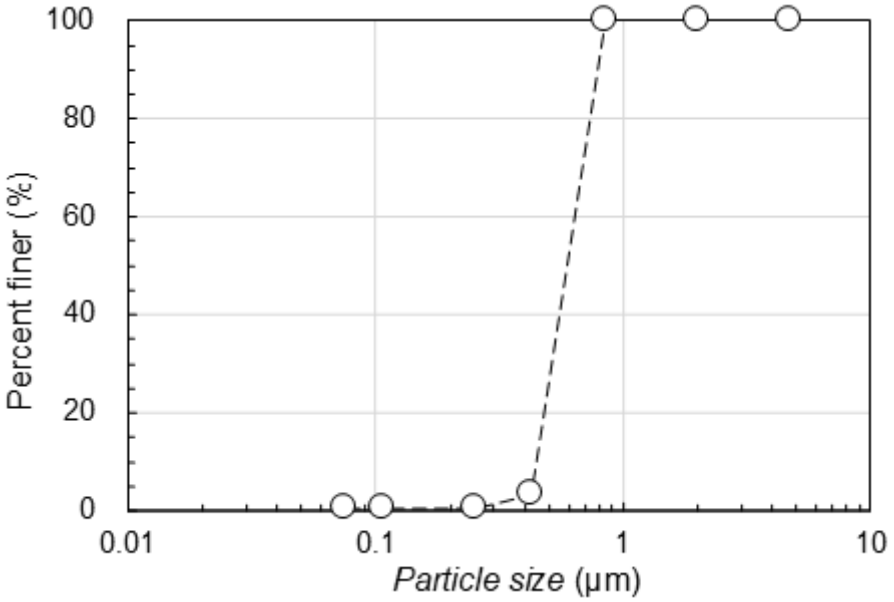

Fig 1. Particle size distribution curve of Jumunjin sand

2.1.3 Jumunjin sand

This study used Jumunjin standard sand purchased from Joo Mun Jin Silica sand Co., Korea. The particle size distribution (Fig. 1) indicates a mean particle size (D_{50}) of 0.5 mm, a coefficient of uniformity (C_u) of 1.426 and a coefficient of curvature (C_c) of 0.932; classifying it as poorly graded sand (SP) according to ASTM (2006).

2.2 Experiment methods

2.2.1 Preparation of specimens

To investigate the effect of AG and XG ratio, the AG-to-total biopolymer mass ratio (m_a/m_b) was varied as 0, 25%, 50%, 75% and 100%. Additionally, to examine the influence of the total biopolymer content, the biopolymer-to-soil mass ratio (m_b/m_s) was set at two levels: 0.5% and 2%.

Biopolymer powders, premixed when both biopolymers were used, were gradually added to boiling deionized water (DI) produced using water purifier (New Human RO, Human Corporation, Republic of Korea) and thoroughly mixed using a hand mixer to fully dissolve biopolymers. The water content (w) was adjusted to enhance workability: w = 25% for the 2% m_b/m_s and w = 20% for the 0.5% m_b/m_s. The prepared biopolymer solution was then uniformly mixed with the soil, ensuring the temperature remained above 70 °C to premature gelation of AG (Jang et al., 2024). Subsequently, the biopolymer-treated soils were placed into cubic molds measuring 4.5 cm per side. Specimens were dried at room temperature (22 ± 2 °C) and a relative humidity of 50% for periods of 1, 3 and 7 days. Detailed specimen descriptions are summarized in Table 1.

Table 1. Specimen preparation conditions varying biopolymer contents, crosslinking ratios, and drying/submerging periods

Biopolymer-to-soil mass ratio, m_b/m_s [%]	AG-to biopolymer ratio in mass, m_a/m_b [%]	Water content, w [%]	Drying time [d]	Experimental condition
2	0	25	1	Dried only
	25			
	50		3	
	75			
	100		7	
0.5	0	20	3	1d Submerged
	25			
	50		7	1d Submerged
	75			
	100			Dried only

2.2.2 Uniaxial compressive strength (UCS)

The uniaxial compressive strength (UCS) of specimen was measured with DA-507 testing machine (Dong Ah Testing Machine, Republic of Korea) at a constant strain rate of 1 %/min, following the ASTM D2166/D2166M-16 (2016). The effect of m_b/m_s on UCS was investigated using specimens containing 0.5% and 2.0% m_b/m_s after drying periods of 1, 3, and 7 days. Additionally, a separate set of specimens containing 0.5% of m_b/m_s was submerged in DI for 1 day after the initial drying, then re-dried for 7 days to assess the durability against water immersion.

3. RESULT AND ANALYSIS

3.1 Effect of drying period on the UCS of sand

Fig. 2 presents a comparison of the UCS of sand specimens containing a 2% m_b/m_s, treated with different m_a/m_b (i.e., 0%, 50%, 75% and 100%), evaluated after drying periods of 1, 3, and 7 days. A clear and consistent trend emerged, indicating increased UCS values with extended drying durations. Regardless of m_b/m_s, specimens dried for 7 days consistently exhibited the highest UCS, with notable reductions observed in specimens dried for shorter periods (3 days and 1 day). Furthermore, the AG content significantly influenced UCS performance; higher AG proportions generally resulted in reduced UCS. Specifically, after 7 days of drying without water immersion, specimens containing only XG ($m_a/m_b = 0$) demonstrated the highest UCS of 2.1 MPa. In contrast, specimens containing only AG ($m_a/m_b = 1$) showed the lowest UCS of 105.03 kPa.

Additionally, the rate of strength development with drying period was more pronounced in specimens containing higher proportions of XG. This behavior can be explained through the water evaporation ratio (*ER*; the ratio of mass decrease during drying to the initial specimen mass) over time (Fig. 3). A distinct trend was observed in the initial 3 days drying period, where the *ER* positively correlated with the AG content; specimens with a higher m_a/m_b exhibited greater initial water loss. Thus, soils with a

higher m_a/m_b showed a larger increase in UCS between 1-day and 3-day drying periods. After 7 d, however, the *ER* exhibited minimal variation across all compositions, ranging narrowly between 15.08% to 15.51%. These findings indicate that AG-rich specimens dried rapidly within the first 3 days, after which the *ER* decreased significantly. In contrast, XG-rich specimens demonstrated superior initial water retention but continued to lose moisture more steadily throughout the 7d period. Therefore, these findings indicate that XG is more effective for short-term moisture retention, whereas AG is characterized by a higher initial rate of evaporation.

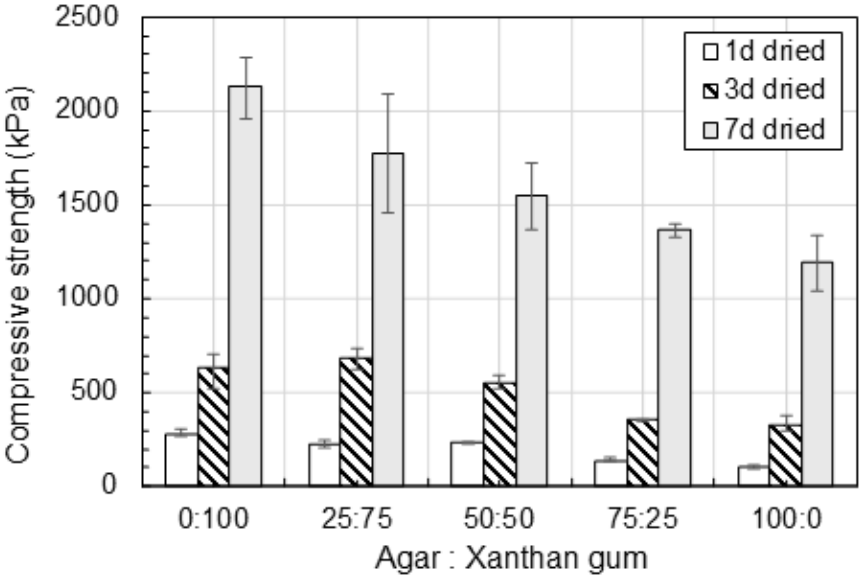

Fig 2. Uniaxial compressive strength (UCS) of specimens treated with various agar-to-xanthan gum ratios after drying periods of 1, 3, and 7 days (biopolymer-to-soil mass ratio, m_b/m_s = 2 %).

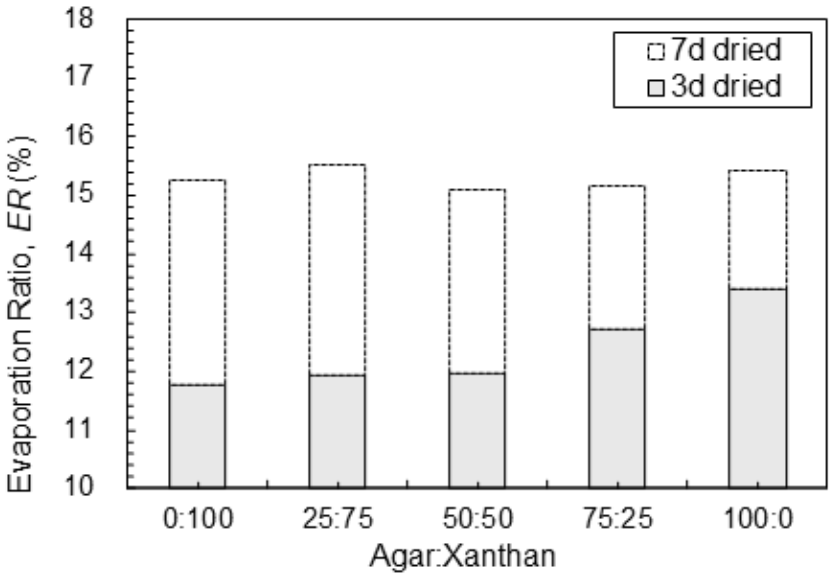

Fig 3. Evaporation ratio (*ER*) of specimens treated with various agar-to-xanthan gum ratios after drying periods of 3 and 7 days (biopolymer-to-soil mass ratio, m_b/m_s =0.5%)

3.2 Effect of total biopolymer content on the UCS of sand

Fig. 4 illustrates the influence of m_b/m_s on UCS values after a drying period of 7 days, comparing specimens containing 0.5% and 2% m_b/m_s. Results clearly demonstrate that UCS values significantly increase with higher m_b/m_s. Pure XG treated specimens with 0.5% m_b/m_s exhibited the highest UCS of 1273.52 kPa among the lower biopolymer content group, yet this strength was considerably lower by 860.34 kPa compared to the corresponding 2% m_b/m_s specimens. Additionally, as the proportion of AG increased, UCS values consistently decreased, emphasizing the lower mechanical performance of AG compared to XG in initial dried states. This comparison confirms that not only the biopolymer type but also the total biopolymer content considerably impacts the mechanical properties and compressive strength enhancement of treated sand.

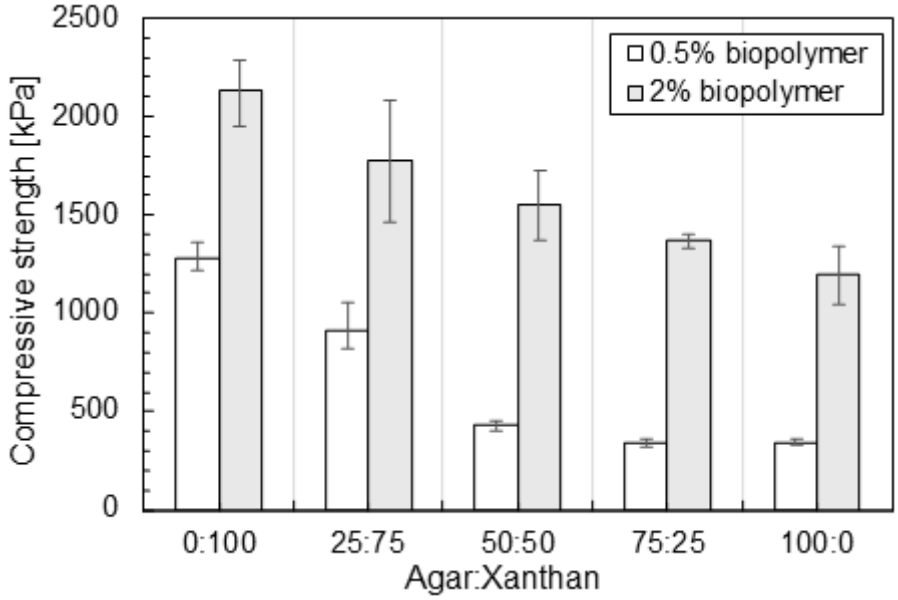

Fig 4. Comparison of uniaxial compressive strength (UCS) between specimens with different biopolymer-to-soil mass ratios: 0.5% and 2%, measured after 7 days of drying.

3.3 Effect of water immersion on the UCS of sand

Figure 5 summarizes the structural integrity of 0.5% total biopolymer-treated soils after being submerged in DI for 1 day. After submergence (Fig. 5a), specimens with a larger AG (i.e., m_a/m_b = 75% and 100%) maintained their intact cubic shape without visible soil loss. In contrast, the specimens having lower AG (i.e., m_a/m_b = 0%, 25% and 50%) lost structural integrity after submersion. Particularly, especially the soil treated by XG more than half of biopolymer content (i.e., m_a/m_b = 0% and 25%) completely lost their initial shape with more than 85% of soil loss occurred (Fig. 5b). For this reason, specimens having XG more than AG were impossible to measure the UCS, indicated 'N/A' in Fig. 6.

The UCS of submerged pure AG (m_a/m_b=1) is 317.41 kPa, which is similar with the same AG content but only dried specimen. As the content of XG increased, the specimen with m_a/m_b=75% showed higher UCS of 500.45 kPa. This result also indicates that water

resistance is not proportionally dependent on the amount of AG. Instead, a specific ratio of XG is required for a higher UCS.

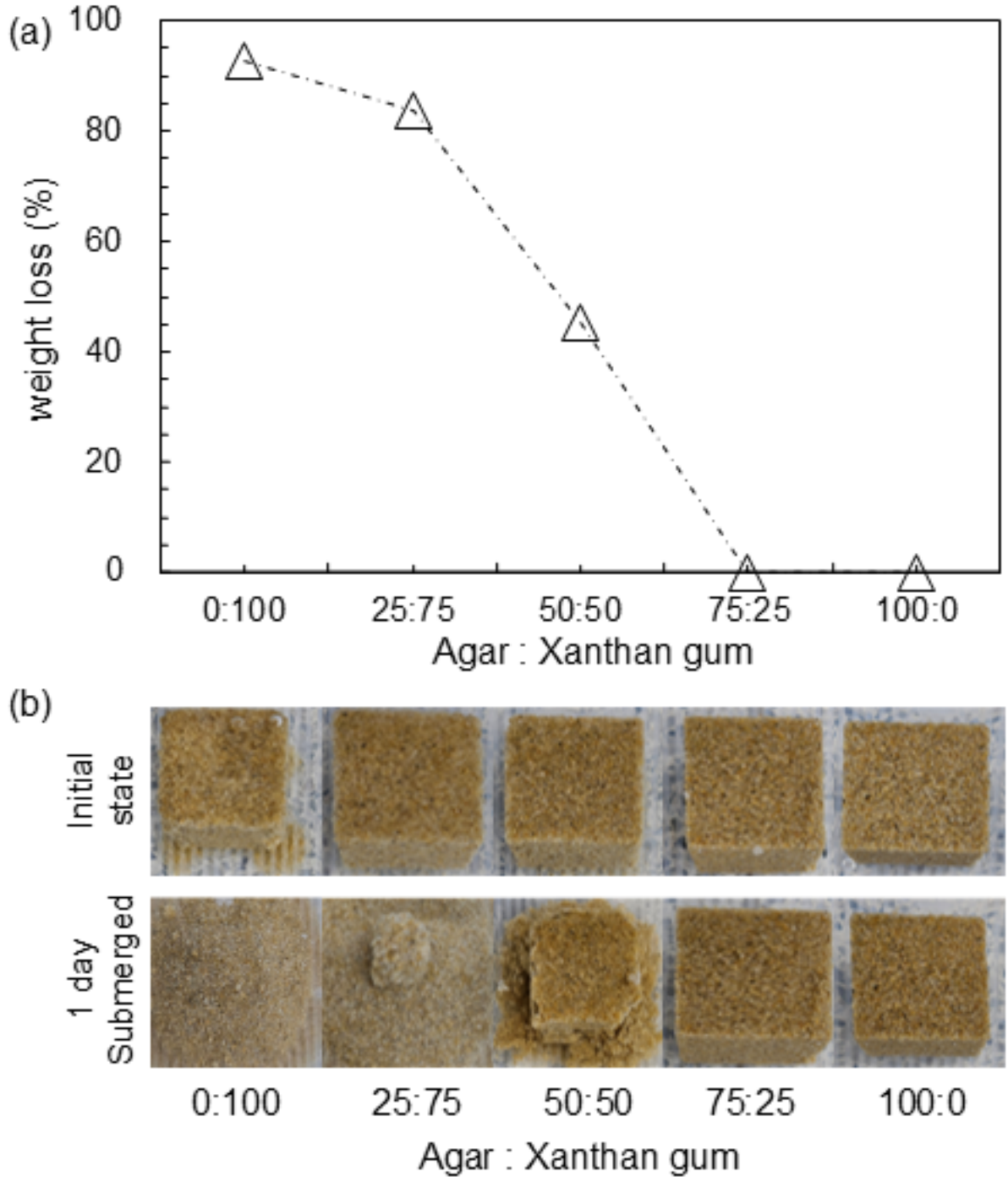

Fig 5. Comparison of soil loss and structural integrity before and after submersion for specimens treated with various agar-to-xanthan gum ratios (biopolymer-to-soil mass ratio = 0.5%). (a) weight loss ratio, (b) optical difference of each soil (i.e., m_a/m_b=0, 25%, 50%, 75% and 100% from the left)

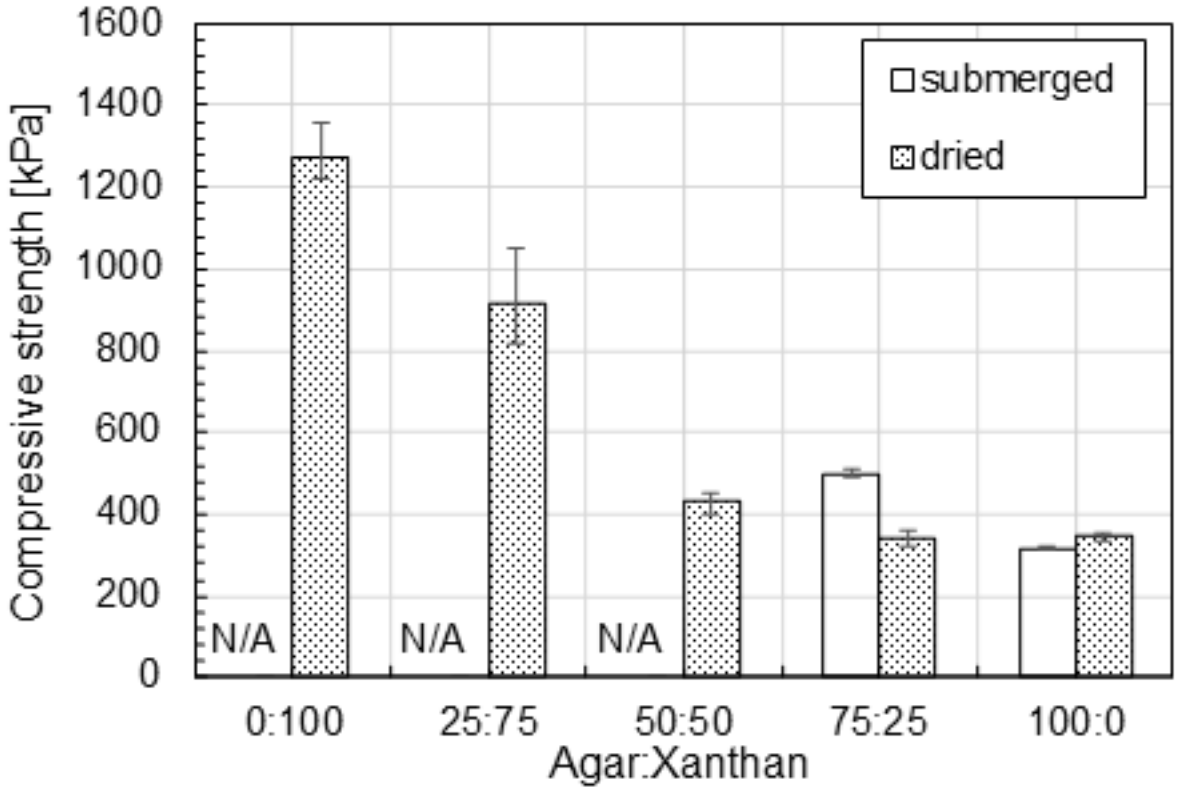

Fig 6. Comparison of UCS values between dried and submerged specimens treated with various agar-to-xanthan gum ratios (biopolymer-to-soil mass ratio = 0.5%). Specimens marked as 'N/A' disintegrated upon submersion, making UCS measurement impossible.

3.4 AG-XG Crosslinking mechanisms

AG forms helical dimer structures composed of 1,3-linked β-D-galactopyranose and 1,4-linked 3,6-anhydro-α-L-galactopyranose, which enable the formation of a rigid gel matrix (Nanna Rhein-Knudsen et al., 2015). Due to its low content of anionic sulfates, AG exhibits relatively hydrophobic characteristics (N. Rhein-Knudsen et al., 2015). This relative hydrophobicity contributes to the ability of Ag-rich sand (i.e., m_a/m_b = 75% and 100%) to retain their shape under water. Furthermore, incorporating a small amount of XG enhances the biopolymer gel by decreasing hydraulic conductivity and increasing water contact angle by forming hydrogen bonding between carboxyl group ($COOH^-$) of XG and hydroxyl group (OH^-) of AG (Rukmanikrishnan et al., 2019). These stronger intermolecular interactions improve the cohesion of the biopolymer matrix, allowing AG-XG treated sand (m_a/m_b=75%) to exhibit higher UCS and to retain structural integrity after submersion. In contrast, specimens with higher XG (m_a/m_b = 0, 25%, and 50%) lose their structural form due to the hydrophilic nature of XG, which disrupts AG gelation.

4. CONCLUSIONS

This study investigated the effectiveness of agar (AG) and xanthan gum (XG) biopolymer crosslinking for enhancing the uniaxial compressive strength (UCS) and

water resistance of sand. A series of UCS tests were conducted on specimens treated with various AG-to-XG ratios (i.e., m_a/m_b = 0-100%) and total biopolymer-to-soil ratio (i.e., m_b/m_s = 0.5% and 2%), under different drying durations and submersion conditions.

The key findings of this study include:

- XG-rich specimens exhibited higher UCS after drying, particularly at higher biopolymer contents (m_b/m_s = 2%). This is attributed to the superior binding capacity and strength development potential of XG in dry conditions.
- AG-rich specimens, although lower in UCS in the dried state, retained their structural integrity upon submersion, owing to the rigid gelation behavior of AG.
- Optimal mechanical performance and water durability were observed in specimens with moderate AG-to-XG ratios (e.g., m_a/m_b = 75%), where intermolecular hydrogen bonding between AG and XG enhanced both strength and resistance to water-induced disintegration.
- Water resistance was not simply proportional to AG content; instead, a specific crosslinking ratio was found to be critical in balancing strength and durability.

These findings highlight the potential of AG-XG crosslinked biopolymers as sustainable grouting agents for geotechnical applications, particularly in environments where both strength and water durability are essential, such as tunnels and underground spaces. However, the current study was limited to short-term submersion conditions. Further research is needed to evaluate the long-term stability and degradation behavior of biopolymer treated soils under prolonged water exposure and field conditions.

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