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Temporary consolidation and excavation of artifacts at waterlogged archaeological sites

Xue-Qiang Chen, Lina Xie✉, Shiqiang Fang, Wenjing Hu & Ming Cao

Ensuring the preservation of fragile artifacts from waterlogged archaeological sites demands the advancement of extraction techniques that uphold both safety and the integrity of historical data. Conventionally employed extraction materials are predominantly applicable to sites exhibiting high soil stability and low moisture content, whereas the recovery of artifacts from waterlogged contexts remains technically challenging. This study explores low-acyl gellan gum as a temporary solidification material for waterlogged archaeological relics, enhancing its efficacy through ionic cross-linking methodologies. The results show that by meticulously regulating the species and concentration of cations—specifically Na^{+}, Mg^{2+}, Ca^{2+}, or Al^{3+}— an improvement in gelling strength can be attained within an optimum range. The gel strength improved with the increase of ion valence, but adding Al^{3+} results in local gel aggregation. By adding 3.0 wt% of $CaCl_2$, low-acyl gellan gum can form a transparent, stable gel with a compressive strength amplified to 0.11 MPa. In addition, the temporary consolidation and extraction experiment under laboratory conditions shows that Ca^{2+} cross-linked gellan gum can effectively carry out temporary solidification and safe extraction of multiple artifacts at waterlogged archaeological sites. This innovation presents a simplified yet potent strategy for excavating fragile artifacts.

Keywords Waterlogged archaeological sites, Low-acyl gellan gum, Crosslinking, Temporary consolidation, Excavation of artifacts

Cultural relics that have been buried for a long time are corrupted by the surrounding complex environment's physical, chemical and biological effects, and cause the integrity of cultural relics is difficult to preserve. After excavation, the buried environment was disturbed, and the safe extraction of archaeological excavation sites is the basic work to ensure the physical condition, restoration, and long-term preservation. The original state of artifacts on the archaeological site contains important historical and cultural information, and the complexity of its surrounding environment makes it difficult for archaeologists to obtain complete information in a short time.

The archaeological excavation process adheres to the principle of minimal intervention, aiming to preserve the original state of the cultural relics to prevent affecting the authenticity[1]. Excavation by extraction, which entails unearthing artifacts along with the encompassing soil, ensures the preservation of fragile specimens in situ. This approach constitutes a safeguard for their secure retrieval. Given the fragility of these artifacts, employed solidifying agents must exhibit substantial reinforcing capabilities to withstand exterior physical forces. The cutting method is relying on the strength of the surrounding soil. This method is suitable for the situation where the surrounding soil is in good condition, but it is not suitable for large cultural relics. It is a common method to strengthen the whole cultural relic through gypsum pouring[2]. Due to the large density of gypsum, it is also not conducive to reinforcing large cultural relics. Alternatively, polyurethane foam materials can bolster the peripheral soil strata and are frequently utilized for reinforcing voluminous artifacts. However, this foam material is difficult to reinforce in a low-temperature environment, and the flammability of the material increases its safety hazards.

Moreover, sublimable substances can serve as transient stabilizers, with their sublimation property ensuring a reversible process. Through melting and re-curing, cyclododecane and menthol have emerged as prevalent temporary consolidants for fragile artifacts[3]. Hangleiter used cyclododecane as a temporary curing agent for archaeological sites, while Brown effectively utilized it to fortify delicate lizard fossils, facilitating a secure transit from Chicago to New York[4,5]. As one of the earliest extraction materials to be widely adopted, it has found extensive application despite certain limitations, such as its high melting point and potential toxicity to human health and the environment[6]. In recent years, menthol has been gradually applied in archaeological sites due

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to its higher operating safety, suitable melting point and reinforcement performance. Menthol has been used for on-site extraction of Qin terracotta pit painting and armors[6]. Experimental comparisons conducted on the reinforcing efficacy of quartz sand, carbon powder, and oak powder, each with varying moisture levels, revealed that the inherent hydrophobic nature of cyclododecane and menthol impedes their ability to wet waterlogged artifacts' surfaces, thereby impinging on consolidation efficiency. Consequently, the materials above are only suitable for archaeological sites with low moisture content[7]. However, a large number of artifacts unearthed in waterlogged archaeological sites have endured prolonged erosion, encompassing fragile relics such as lacquered wood, silk fabrics, bamboo, bone and stacked artifacts. These finds are in a precarious state, rendering their direct extraction via conventional methodologies a challenge. Therefore, given the diversity of cultural relics materials and the surrounding environment in the site, the preservation situation is complex, and it is urgent to develop special archaeological extraction materials and technical methods suitable for waterlogged sites[8,9]. Utilizing polymer bandages, Xia et al. recently accomplished the successful extraction and preservation of elephant tusks in a water-saturated state, which were unearthed from the No. 3 sacrificial pit of the Sanxingdui site[10]. The successful implementation of the aforementioned case demonstrates the practical utility of polymer bandages as a material for extracting artifacts in humid archaeological environments. It is worth noting that such polymer bandages have been previously employed in the extraction of underwater cultural heritage[11]. The process of applying bandages necessitates the movement of artifacts, which may potentially compromise the integrity of their in-situ archaeological context and preservation status.

Low-acyl cold adhesive is a polymer material, also known as low-temperature cold adhesive or hot melt adhesive. It exhibits a solid form at room temperature, and it can be turned into a homogeneous liquid state when heated to a certain temperature. Cooling hot solutions can produce transparent, hard and brittle gels at a low polymer concentration. The involved gelation mechanism is a coil-helix transition followed by lateral helical aggregation[12]. This remarkable gelling capacity allows the wide use of low-acyl gellan gum (GG) in the food industry as thickening, gelling and structuring agent[13,14]. Besides, low-acyl GG has outstanding advantages in flexibility, biocompatibility, degradability and acid stability[15]. At present, gelling gum has been widely used, such as food[16], medical[17], wastewater treatment[18], flexible electronics[19] etc. It can be spontaneously gelated at room temperature, which is thermally reversible[20]. However, using single type of gellan gum may present limitations, such as poor mechanical properties, high gelling temperature, and instability in physiological conditions, which cannot meet the requirements of wide or special applications. Previous results have shown that, gellan gum can transform into harder (rigid) with greater thermal stability and optimal gelling conditions[15,21–23]. Cations can unite with carboxyl groups in the molecular chain to enhance the force between molecular chains and thus improve the strength of the gel.

To enhance its mechanical attributes and facilitate its deployment in the provisional solidification of excavated artifacts, ionization and cross-linking of the adhesive are imperative steps. By adding metal cations with different valence states, the ions are evenly dispersed into the molecular chain of gellan gum to form a clear solution, which is further cooled to room temperature to form a stable transparent gel. Similar to cyclododecane and menthol, GG has also been commercialized and is available for purchase through commercial channels. In comparison to the other two, GG is priced more competitively. Additionally, as an edible food additive, GG exhibits a favorable environmental profile.

Based on the structural characteristics and formation mechanism of low-acyl GG, this work aims to investigate its temporary solidification property used for waterlogged artifacts and techniques for enhancing its performance. Our research found that employing a reduced quantity of low-acyl GG successfully generates a stable gel with high transparency and strength. The gel's strength is ingeniously modulated via the manipulation of ion species and their concentrations. In terms of compatibility perspective, the gelation process is gradual and compatible with the waterlogged cultural relics. The experimental results demonstrate that low-acyl GG is more suitable for the solidification and extraction of waterlogged archaeological artifacts compared to cyclododecane and menthol. Additionally, unlike wet-curing bandages used in moist environments, which require the relocation of artifacts, low-acyl GG can complete the extraction process without moving the artifacts. This ensures the integrity of the artifacts' original state is fully preserved throughout the entire process. Furthermore, exhaustive laboratory assessments affirm the promising application prospects of this material, thereby underlining its extensive utility potential in preserving waterlogged artifacts.

Experimental section

Materials

Low-acyl gellan gum (food grade, 99%) was purchased from Shanghai Xintai Industrial Company. NaCl (99.9%), $MgCl_2$ (99%), $CaCl_2$ (99.9%), menthol (99.5%) and cyclododecane (99%) were purchased from Aladdin Biochemical Technology Co., Ltd. Al_2Cl_3 (99%) were purchased from Sinopharm Chemical Reagent Co., Ltd. Deionized water was made in laboratory. No further purification was performed before all chemicals use. Bamboo slips used in the simulated archaeological site extraction experiments are handicrafts (modern samples), and the broken ceramics are ancient celadon fragments (private collection).

Preparation of low-acyl GG gel

GG aqueous solutions of different concentrations (1.0, 2.0, 4.0 and 6.0 wt%) were prepared by adding 2.0, 4.0, 8.0 and 12 g of GG to 200 ml deionized water under magnetic stirring, respectively. The solution was heated to 50ºC with constant stirring until the gellan was evenly dispersed. Low-acyl GG gels were obtained by cool the solution to room temperature. The compressive strength of the prepared gels with different concentrations of low-acyl GG was tested to determine the optimal GG aqueous solution concentration.

The effect of Ca^{2+} percentage on low-acyl GG gel

Low-acyl gellan gum (8.0 g) was dissolved in 200 mL deionized water under magnetic stirring. The solution was heated to 50°C with constant stirring until the gellan was evenly dispersed. Afterwards, different percentage (1.0, 3.0, 5.0 and 10 wt%) of $CaCl_2$ (80 mg, 240 mg, 400 mg or 800 mg of $CaCl_2$ dissolved in 5.0 mL water) was then added slowly under magnetic stirring, respectively. The Ca^{2+} crosslinked low-acyl GG gels were obtained by cool the solution to room temperature. Then, the compressive strength of the prepared gels with different percentage of $CaCl_2$ was tested. And the FTIR spectrums of the gels was measured to obtain the effect of the addition of calcium ions on the low-acyl GG gel.

Compressive strength test of low-acyl GG under the addition of different valence metal ions

The addition of metal cations (Na^+, Mg^{2+} and Al^{3+}) are prepared following the method described by the above process. In briefly, 3.0 wt% of NaCl, $CaCl_2$ or $AlCl_3$ (240 mg dissolved in 5.0 mL water) was added to GG aqueous solution (4.0 wt%, 200 mL) under magnetic stirring, respectively. Cooling to room temperature and the compressive strength of GG gels containing different valence metal ions was tested. The size of the test sample is $2 \times 2 \times 2$ cm.

Characterization

Fourier transform infrared spectroscopic (FTIR) analysis was performed on a Nicolet iS10 (Thermo Fisher Scientific, U.S.A.) spectrometer in the Attenuated Total Reflectance (ATR) mode. And the spectra were recorded in the wavenumbers region from 4000 to 400 cm^{-1} with a resolution of 1 cm^{-1} and 16 scans. The contact angle was measured with an automatic contact angle measuring instrument (Shanghai Fangrui instrument Co., Ltd, JCY series) using a test droplet size of 5 μL.

Results and discussion

The chemical structure, preparation route and gelling mechanism of the low-acyl GG are shown in Fig. 1. According to the chemical structure, gellan is a linear anionic polysaccharide consisting of repeating units of tetrasaccharide sequences, including two β-$_D$-glucose residues, one β-$_D$-glucuronate residue and one α-L-rhamnose residue (molar ratio of 2:1:1)[24]. Low-acyl GG gels exist in the state of irregular coil in hot solution and form double helix after cooling, which will aggregate in the presence of cation, and form a network structure through hydrogen bond, molecular chain entanglement interactions, making it have good gelation and film forming characteristics.

As the concentration increases, the mechanical properties of the GG gels are observed to enhance. Figure 2 illustrates that during the preparation of low-acyl GG solutions at varying concentrations, full dispersion is still achievable even upon reaching a 4.0% equivalence ratio. In cooling to room temperature, uniform and stable gels can be gradually formed. Nonetheless, as concentration escalates from 1.0 to 6.0%, complete dissolution of gellan gum into the aqueous medium becomes increasingly challenging. The emergence of insoluble clumps hampers the subsequent gelation process upon cooling, leading to visibly flawed gels. Consequently, employing a 4.0% equivalence of low-acyl GG for gel fabrication emerges as a prudent strategy.

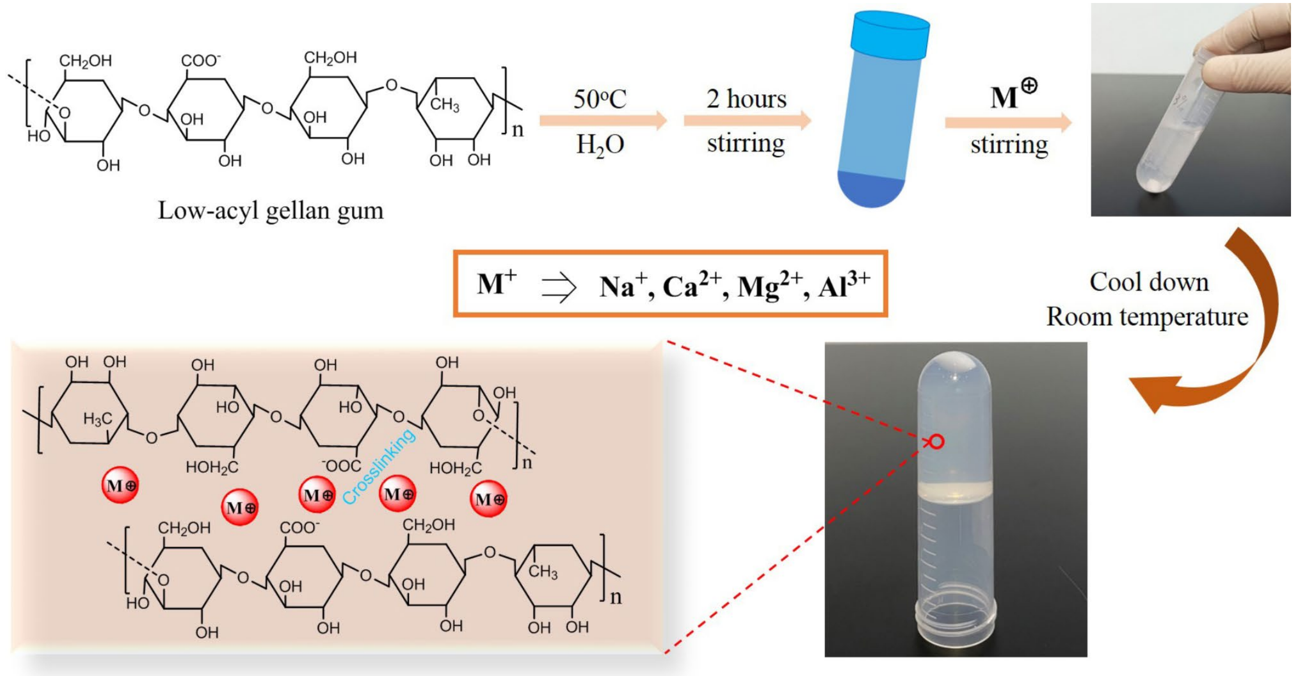

Fig. 1. Schematic illustration of preparing ion crosslinked low-acyl GG.

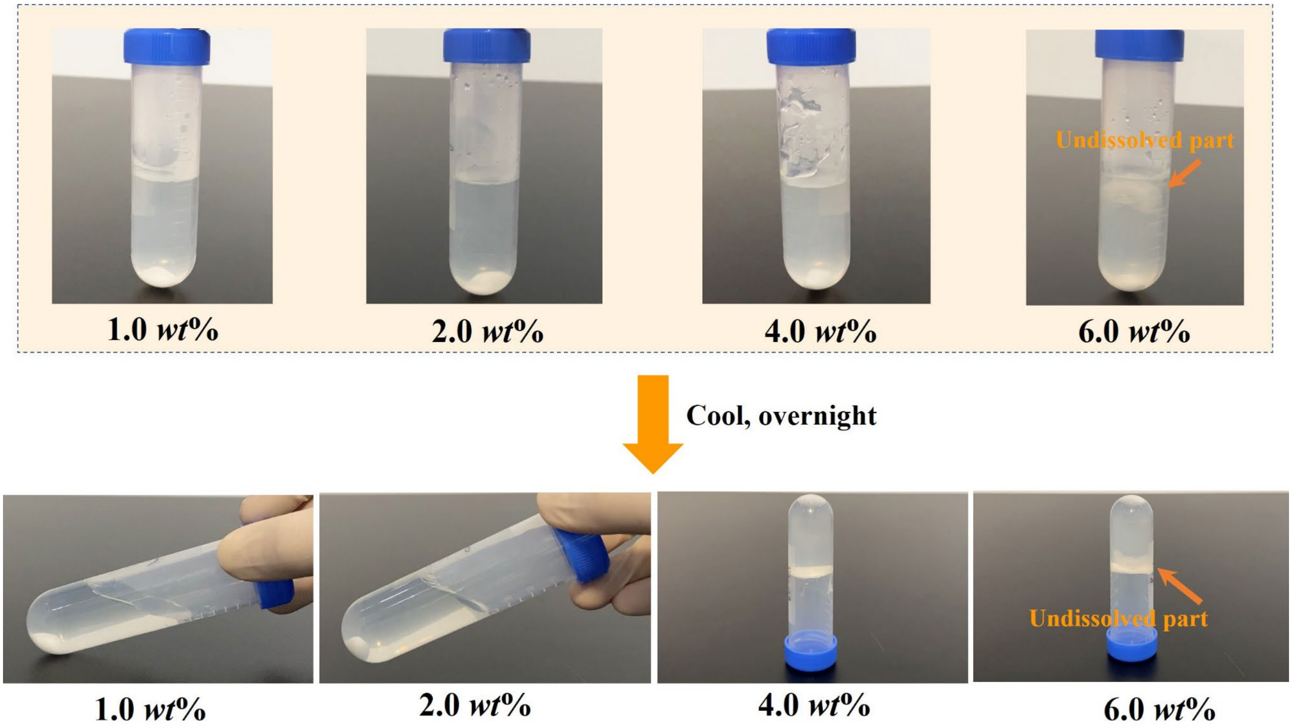

Fig. 2. State images of the dispersive liquid with different mass fractions of low-acyl GG (1.0, 2.0, 4.0 and 6.0 wt%) before and after cooling.

Specifically, the inclusion of cations such as Ca^{2+} results in the genesis of a firm, crystalline-like gel exhibiting a stable and orderly structure. As shown in Fig. 3a, different dispersion states were observed after adding different equivalent ratios of $CaCl_2$ (1.0, 3.0, 5.0 and 10.0 wt%) to the dispersed solution of low-acyl GG. The introduction of lower concentrations of $CaCl_2$ (1.0 and 3.0 wt%) did not affect the dispersion state. However, the introduction with too high concentration (5.0 and 10.0 wt%) will instantly produce flocculent gel due to the aggregation of gel caused by too high local Ca^{2+} concentration. Therefore, considering the required gel's uniformity requirement for the cultural relic solid type, the addition equivalent of $CaCl_2$ should be less than 3.0%. As shown in Fig. 3b, after adding 3% $CaCl_2$, the low acyl gum is still uniformly dispersed and fluid, further cooled to room temperature to form a transparent stable gel. The translucency of the gel ensures real-time visibility of the artifact's condition, thereby guaranteeing the artifact's safety throughout transportation.

In addition, by measuring the compressive strength of the gel after adding different equivalents of $CaCl_2$, it is found that with the increase of Ca^{2+} concentration, the gel strength increases significantly (Fig. 4a). The interplay between low-acyl GG and Ca^{2+}concentration are two mutually influencing parameters, which can be used to control the optimal gel hardness and uniformity. Further tests indicate that GG gels can maintain stability over extended periods. Additional temperature elevation experiments show that, at room temperature, GG gels can maintain their strength stability until the temperature rises to 75 °C (Figure S1). Remarkably, when a weight of 1 kg is applied onto the ion-crosslinked low-acyl GG (containing 3.0% Ca^{2+}) after adding ions, which can still maintain its initial configuration exhibits no significant deformation, showing excellent resistance against external interference (Fig. 4b). To delve into the molecular interactions underlying these changes, Fourier Transform Infrared (FT-IR) spectroscopy was employed. Figure 4c showing the FTIR spectra of low-acyl GG with different amount of $CaCl_2$. In case of pure low-acyl GG, absorption peak at 3334 cm^{-1} is attributed to the presence of H-bonded O-H stretch vibration of hydroxyl groups, and the weak absorption peak at 2923 cm^{-1} is the C-H stretch vibration of CH_2 group. The peak of 1608 cm^{-1} is attributed to asymmetric carboxylate anion stretching (-C=O). Symmetric carboxylate anion and C-O stretching were observed at 1409 and 1016 cm^{-1}. In the spectrum of GG with different proportions of $CaCl_2$, the absorption peak of the asymmetric carboxylate anion group (-C=O, 1608 cm^{-1}) splits into shoulder peaks. These alterations potentially arise from carboxyl group crosslinking with Ca^{2+} during the ionotropic gelation process[25].

Being inherently anionic polysaccharides, gellan is sensitive to the presence of metal cations, with the gelation process being significantly influenced by the ionic species and strength. Monovalent cations are believed to promote the coil-helix transition and the helical aggregation by exerting an electrostatic shielding effect[26]. In contrast, divalent cations directly bind the carboxylate ions on different GG chains and more effectively promote the coil-helix transition and the aggregation of double helices. Until present, little is known about the influence of trivalent cations on gelation of gellan[27]. To optimize gel performance, we ventured into examining the regulatory impact of introducing varied valency cations on low-acyl GG. As shown in Fig. 5, gels of appreciable strength can be attained with low-acyl GG using not only monovalent cations like sodium but also divalent cations such as magnesium and calcium, and even trivalent aluminum ions. At the same time, the gel strength

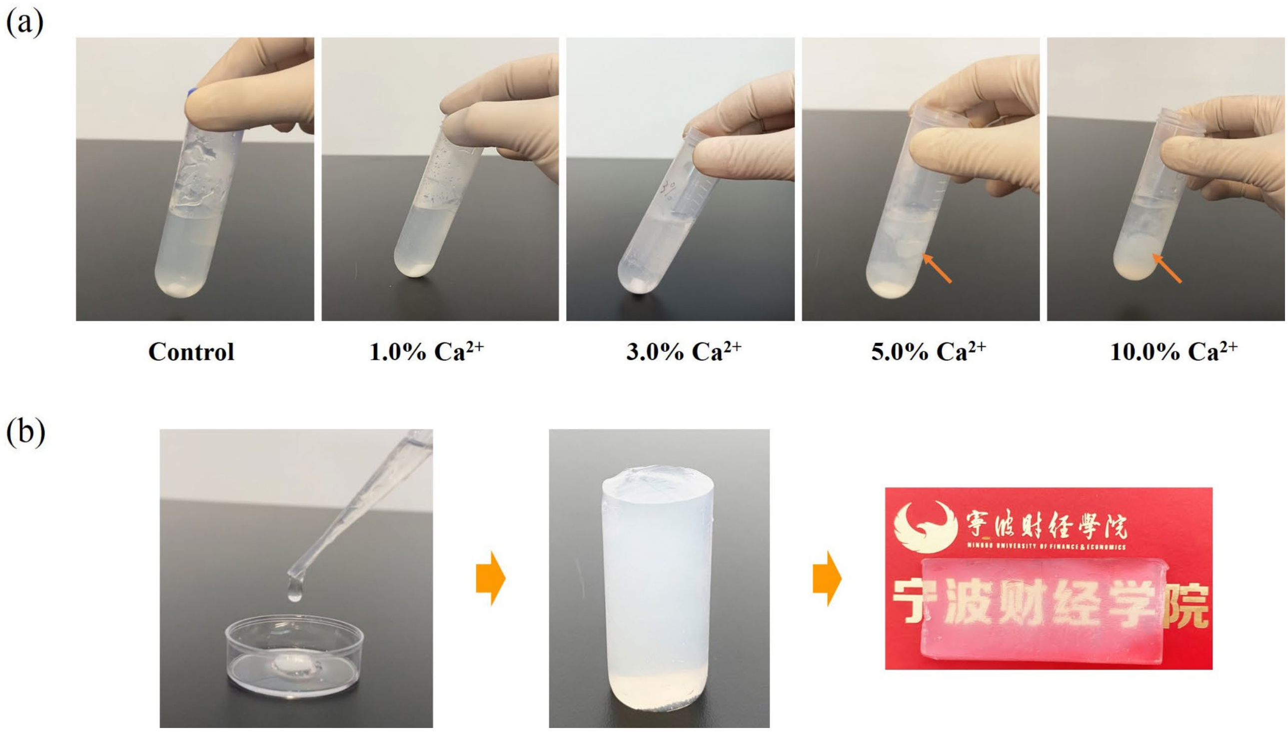

Fig. 3. (**a**) Effect of different adding ratio of $CaCl_2$ on dispersibility of gellan gum in water. (**b**) The state of gellan glue before and after cooling (3.0% $CaCl_2$).

increased with the valence state of the added metal cations, especially the improvement effect from monovalent ions to divalent ions. Whereas calcium or magnesium introduction yields gels of superior strength, sodium-ion-mediated gelation produces gels of relatively low strength. Remarkably, the inclusion of trivalent ions also markedly bolsters gel strength, outperforming the efficacy of divalent ions. Nonetheless, after the addition of aluminum ions, the phenomenon of local gelation quickly occurs in the gelling gel dispersion liquid system, diverging from the behavior observed with calcium or magnesium ions. It results in a thin-film colloidal gel that is challenging to disperse. To strike a balance between maximizing gel strength and forestalling premature localized gelation, the strategy of incorporating 3% soluble calcium salts during the hot phase is advocated. This practice ensures the formation of a homogeneous gel upon cooling, thereby preventing the formation of undesirable local colloidal gels.

Under the condition that the surface of artifact is soaked in water, the wettability of temporary solid materials is important for the reinforcement performance. Due to the surface tension between water-saturated cultural relics and hydrophobic materials, it is difficult for the reinforcement to spread on the surface, which will have a bad impact on the curing process. As shown in Fig. 6, the contact angles on the surfaces of cyclododecane and menthol commonly used in the solidyfied are 101.5º and 56.8º, respectively. Cyclodecane shows obvious hydrophobicity. The contact angle on the surface of low-acyl GG is 38.6º, exhibiting superior hydrophilic properties[28]. Therefore, in comparison to cyclododecane and menthol, low-acyl GG has excellent hydrophilicity characteristics, rendering it more suitable for the reinforcement and extraction of waterlogged artifacts.

To assess the practical performance of ion-crosslinked low-acyl gelatin, we carried out extraction trials on various artifact types at waterlogged archaeological sites. To more accurately simulate the conditions of a waterlogged archaeological site, we conducted extraction experiments in a fully saturated environment. The artifacts and surrounding soil were pre-soaked for a long time, and the entire experimental process was maintained in a continuously water-saturated state. We selected three distinctively layered bamboo slips, semi-submerged in clay, as our extraction subjects. If it is extracted directly without solidification, it will not only break its three-dimensional stacking structure, but also damage the fragile artifacts. As shown in Fig. 7, preliminary to solidification, it is essential to overlay a cubic PVC mold onto the artifacts. Following dispersion and calcium-ion crosslinking at 50 °C, the low-acyl GG is syringed into the mold. However, both menthol and cyclododecane, two commonly utilized extraction materials, require melting prior to use, posing similar challenges when applied at archaeological sites. For sites equipped with an electrical supply, it is feasible to employ a heated magnetic stirrer for on-site sample preparation. Conversely, for locations devoid of electricity, an academically sound approach involves pre-preparing the samples and storing them in a thermos flask before transporting them to the archaeological site for subsequent operations.

As the temperature drops to room temperature, the ion cross-linked gel gum gradually solidifies, forming a transparent and homogeneous gel with high strength. Finally, the waterlogged artifacts can be safely extracted by retaining the pristine state. GG gels demonstrate a robust, jelly-like consistency, distinguishing them from small-molecule compounds such as cyclododecane and menthol, which exhibit prolonged solidification times.

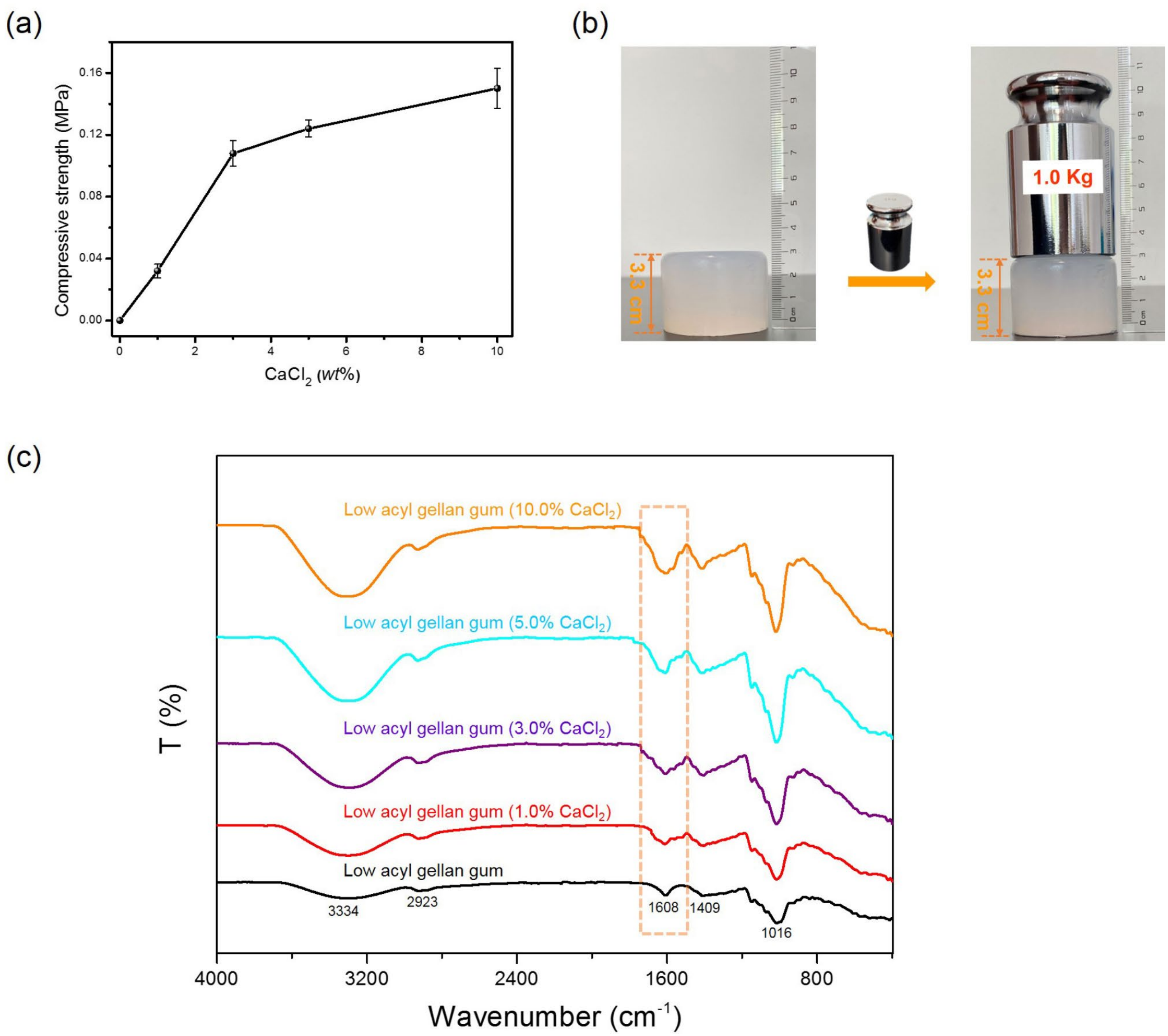

Fig. 4. (**a**) Effect of different addition ratio of $CaCl_2$ on the compressive strength of gellan gum. (**b**) Deformation of low-acyl GG before and after applying weight pressure. (c) FTIR spectra of low-acyl GG with different amount of $CaCl_2$.

These small molecules are capable of permeating into the internal structure of artifacts and necessitate elevated temperatures for sublimation and subsequent removal. In contrast, GG gels undergo rapid polymerization, achieving near-instantaneous solidification. This property enables the safe and efficient removal of the gel through physical detachment methods, and the conceptual diagram illustrating the entire operation process is presented in Fig. 8. Furthermore, this methodology was successfully replicated to accomplish comprehensive solidification and extraction procedures for stacked of broken ceramics, further validating its versatility (Figure S2).

Conclusions

This study delves into the solidification attributes of waterlogged archaeological artifacts, grounded in the architecture and gel properties of low-acyl GG. The results show that the GG capability at reduced dispersion concentrations via cooling processes. Notably, dispersion concentration, ion valence, and incorporation ratios markedly influence the gel's mechanical resilience and homogeneity. While the introduction of higher-valence ions markedly bolsters gel strength, aluminum cations (Al^{3+}) induce localized gelation, hindering subsequent gel progression. After analysis, it is found that a 3.0% calcium ion addition optimizes overall performance enhancement. Considering the challenge of hydrophobic materials in wetting waterlogged artefacts' surfaces, contact angle assessments were carried out. Cyclododecane and menthol (two consolidants commonly used in archaeology) have obvious hydrophobicity, while low acyl GG has super-hydrophilic properties. Finally, the solidification and extraction experiments for various cultural relics at waterlogged archaeological sites show

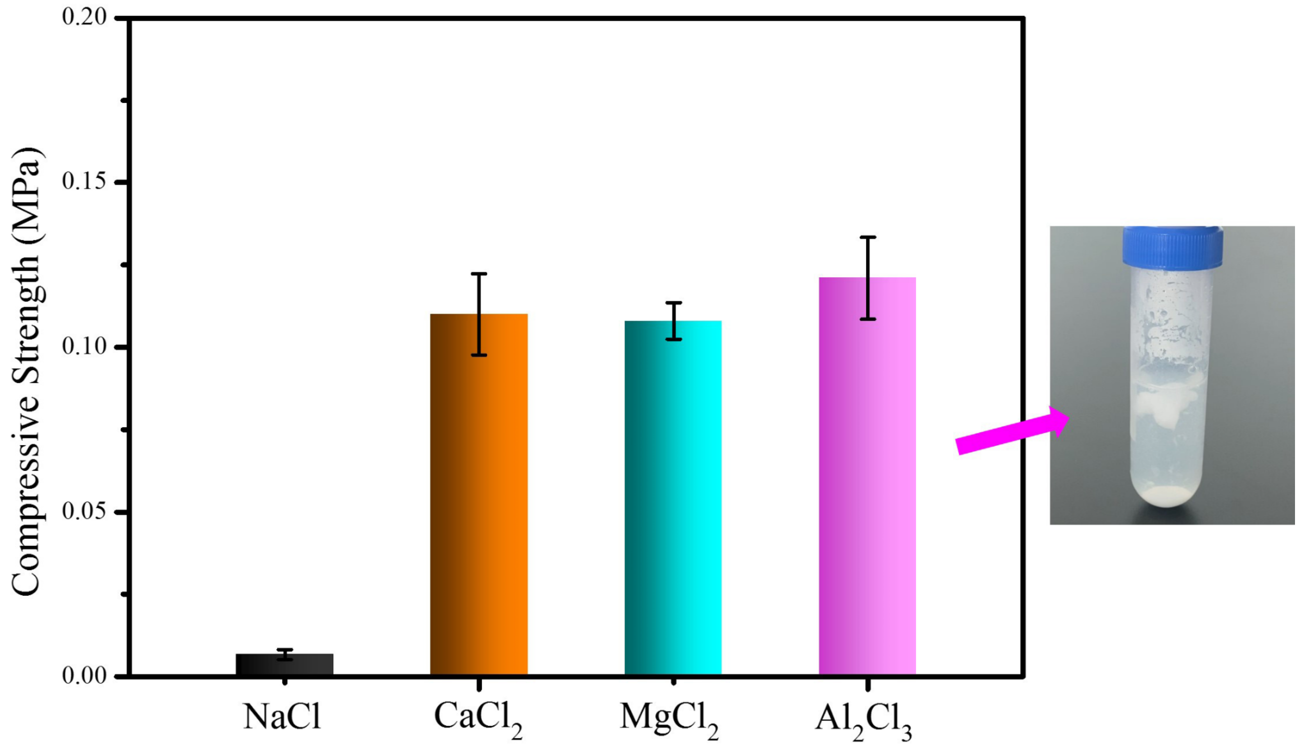

Fig. 5. Compressive strength of low-acyl gel gum under the control of different valence metal ions (Na^{+}, Ca^{2+}, Mg^{2+} and Al^{3+}).

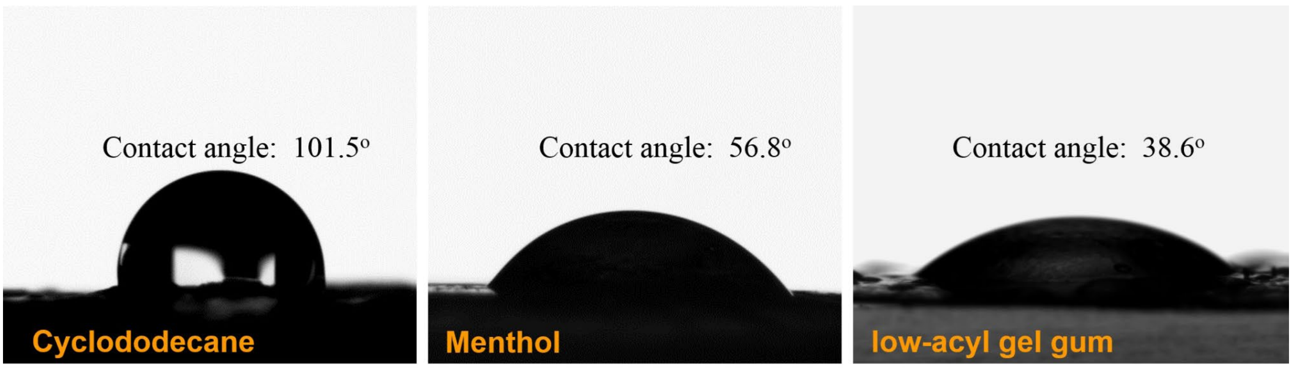

Fig. 6. Water contact angle on the surface of cyclododecane, menthol and low-acyl GG. The data were obtained on the surfaces of cyclododecane, menthol (melted and re-solidified), and gellan gum film (powder dissolved in aqueous solution followed by solidification).

that these materials have promising and remarkable applicability. Nevertheless, several challenges may arise when scaling up for large-scale applications: First, the material exhibits limited applicability in high-temperature environments, such as archaeological sites, where elevated temperatures can impede the curing process and adversely affect the mechanical strength of the consolidated material. Second, its suitability is restricted for the solidification and extraction of certain types of cultural relics, particularly large-volume artifacts, which necessitate consolidating materials with significantly higher mechanical strength.

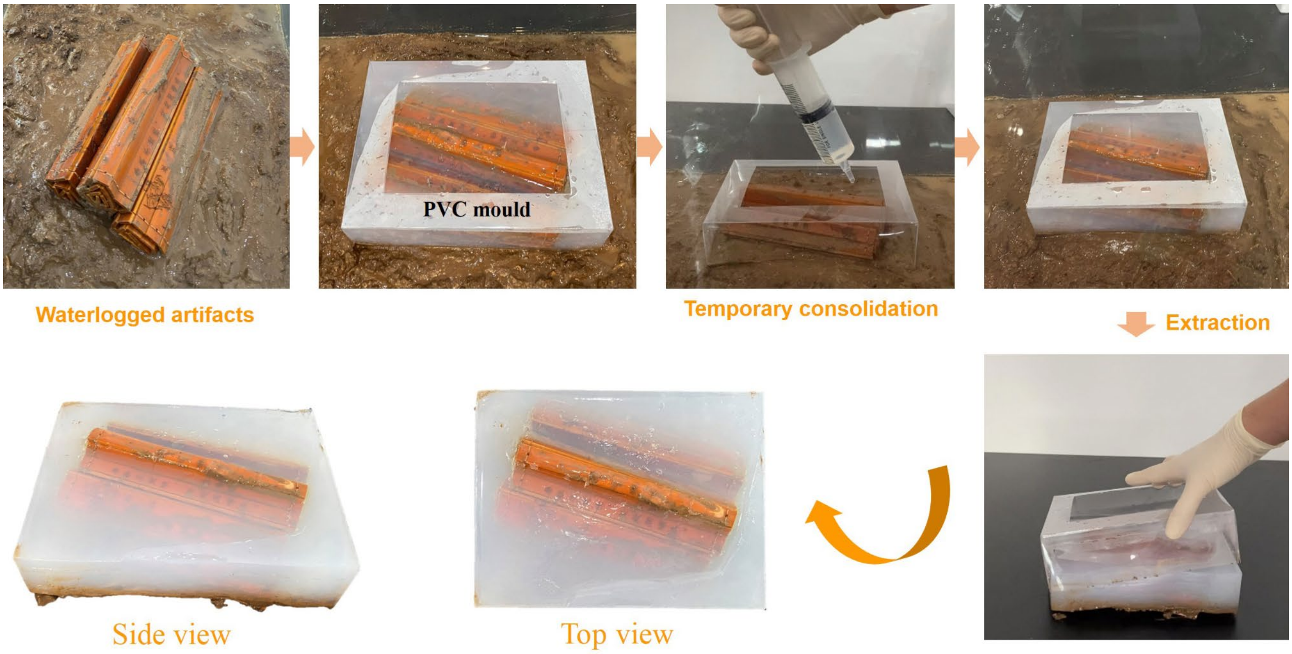

Fig. 7. Process of temporary consolidation and excavation of artifacts at waterlogged archaeological sites. To facilitate the solidification process, a PVC mould is placed over the artifact to be extracted, with openings on top for the addition of gellan gum curing agent. Gellan gum (containing 3% $CaCl_2$) is then injected into the mould using a syringe. Upon contact with the artifact and surrounding soil, the gellan gum rapidly solidifies, forming a transparent and continuous solid matrix. Once fully cured, the solidified artifact is extracted as a whole. The extracted artifact should be maintained in a waterlogged state, and the gellan gum is subsequently removed through physical detachment.

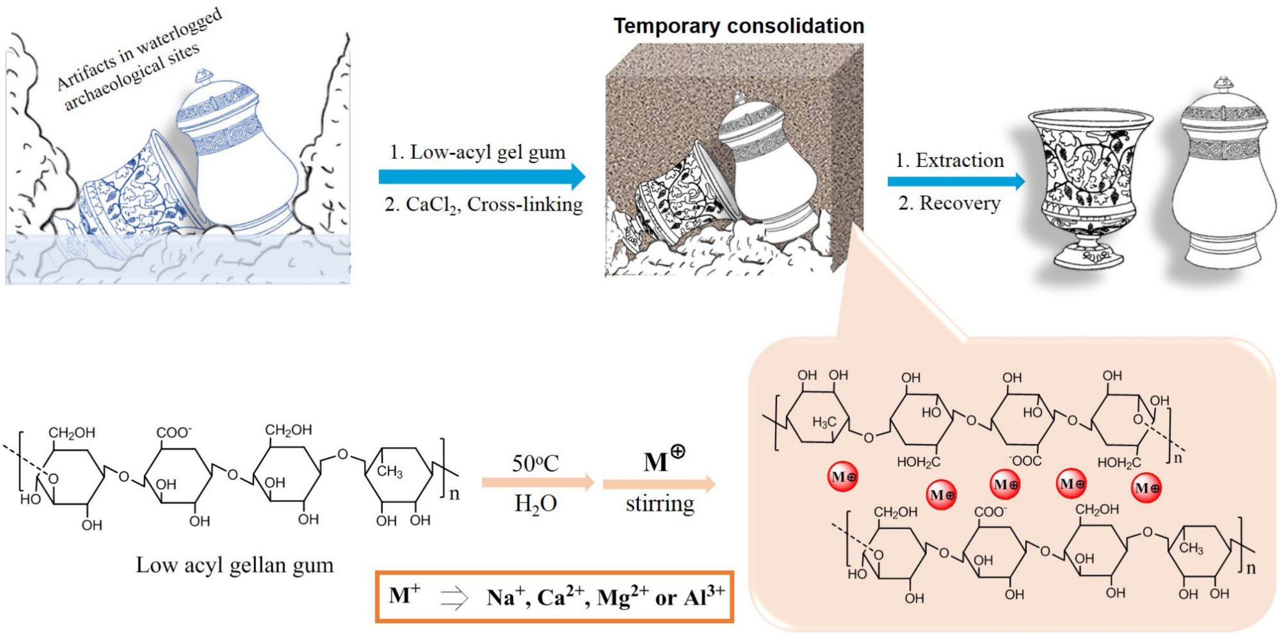

Fig. 8. The conceptual diagram of the temporary consolidation and excavation of artifacts at waterlogged archaeological sites.

Data availability

The datasets used during the current study available from the corresponding author on reasonable request.

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Author contributions

X.-Q.C.: Investigation, writing, conceptualization, L.X.: project administration, writing-reviewing and editing, S.F. and W.H.: conceptualization and editing, M.C.: conceptualization, validation and editing. All authors read and approved the final manuscript.

Declarations

Competing interests

The authors declare no competing interests.

Additional information

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