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Laboratory research of solvent-assisted menthol sols as temporary consolidants in archaeological excavation applications

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Abstract

Temporary consolidation is a quite common practice during archeological excavations nowadays. Melts of cyclododecane or menthol are among the most used consolidants. When using melting protocol to consolidate fragile heritages in archaeological excavation sites, one of the most frequently encountered challenges is the poor penetration of the melt into the heritages, especially in cases of low environmental temperatures, high water content or compact substrates. In this work, we explore the possibility of using solvent-assisted menthol sols as temporary consolidant. Six common organic liquids are individually introduced into menthol at a concentration of 9.1 wt% to formulate room temperature menthol sols. Their potentials as temporary consolidants are systematically investigated. Experimental data indicate that solvent polarity is the most important feature for temporary consolidation purpose and ethanol with medium polarity is among the most appropriate solvents. Laboratory research results show that much better penetration behavior and good consolidation performances can be achieved in menthol-ethanol sol. The as-prepared menthol-ethanol sol is applied in Liangzhu archeological excavation site with satisfactory outcomes. This work shows that menthol-ethanol sol is an excellent temporary consolidation material for archaeological excavation purpose especially in extremely wet condition.

Keywords: Temporary consolidation, Archaeological excavations, Menthol sols

Introduction

Due to their easy removal via sublimation, the use of temporary consolidation materials, also referred as volatile binding media, such as cyclododecane (CDD) [1–9] and menthol [10–14], have considerably increased in archeological excavations. Generally, the materials are applied in their melt form by brushing or dripping. Consolidation is achieved upon solidification of the melts [15–17]. Melting protocol is well accepted because no sophisticated tools are required. Moreover, since there are no other chemicals except binder itself involved, conservators do not need to consider additional possible negative impacts from other chemicals [18].

However, melting protocol has some obvious shortcomings. First of all, some certain heating apparatus and energy source are needed which sometimes may not be convenient on-site especially when archaeological excavation sites are far away from cities [19]. Secondly, previous studies show that best consolidation performances can be obtained when the temperature of the melts is 20–40 °C higher than their melting temperatures [14]. Such high temperature may cause damages to fragile or temperature-sensitive cultural heritages. Recent study also revealed that the internal stress generated in the cultural heritages during melt consolidation is positively related to the temperature of the melts [20–21]. Thirdly, probably more important, it is difficult

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for consolidant melts to penetrate into the heritages under some conditions, which limits their applications. A decent penetration of consolidant molecules into the heritages is essential for consolidation purposes. When the surrounding environment temperature is too low, for instance, winter season in northern China, the melts will solidify too fast to achieve good penetration. Compact or damp substrates are also challenging. Compact substrates have lower pore volume and smaller pores, which are not friendly for consolidant to penetrate. In damp substrates, water prevents consolidant molecules from entering into voids not only due to water molecules occupying the space, but also due to the hydrophobic nature of CDD or menthol molecules [4]. Sprays are another common application form of temporary consolidants and they are easy to carry and use [14, 22]. But sprays also encounter the penetration issue as the melts.

In this work, we examined the possibility of using solvent-assisted menthol sols in temporary consolidation [2, 6, 23]. Organic solvents can arouse safety concerns to the cultural heritages, the environment and human beings [24]. In order to minimize the influence of the organic solvents, the amount of solvent applied in this study is designed to be minimum. In this sense, the materials are referred as sols not solutions. The penetration depth of menthol sols, their binding abilities after solidification, their removal by sublimation and the reverse migration of menthol molecules at various mockup samples are systematically investigated. The as-prepared menthol ethanol sol was successfully applied in Liangzhu ancient city (3300BC– 2300BC), one of the most important Chinese Neolithic culture near Yangtze River Basin [25–27].

Experimental

Materials

L-menthol (>99%, melting point 43 °C, vapor pressure 0.106 kPa at 20 °C) was purchased from Aldrich, used as received. All other chemicals were AR grade, obtained from Sinopharm Chemical Reagent Co., Ltd and used as received, unless stated otherwise.

Four mockup samples were used. Two ISO standard sand (30–40 mesh and 40–60 mesh) were purchased from Xiamen ISO Standard Sand Co. Ltd. Two soil samples, one is Kaolin from Colorobbia Co. (Kunshan); the other soil sample was acquired from one archaeological site of Liangzhu. Both soil samples were sized using 40 and 60 meshes. Soil between 40 and 60 mesh was collected and used. Four mockup samples are named as Sand60 (40–60 mesh), Sand30 (30–40 mesh), Kaolin and Soil-LZ. All samples were dried at 110 °C for 4 h before usage.

The field experiment was carried out at Liangzhu ancient city. Soil reinforced with bamboo mat was found at the ancient river course in Zhongjiagang at Liangzhu. The soil samples were saturated with water. The purpose of the work is to temporarily consolidate bamboo mat reinforced soil, extract and transport it to the laboratory for further studies.

Preparation and characterization of menthol sols

Menthol and the selected solvent were mixed in a round bottom flask. They were heated to a clear solution in a 50 °C water bath. Then the solutions were cooled to room temperature to give out a clear menthol sol. Six common organic solvents were used: methanol, ethanol, 1-propanol, 2-propanol, *n*-hexane and cyclohexane. Some basic physical properties of these solvents are summarized in Table 1. In Table 1, polarity is used to describe the electrical charge distribution over the atoms joined by the bond. Generally, menthol will have stronger interactions and larger solubility with more polar solvent. Boiling point and saturated vapor pressure are related the solvent evaporation. The lower the boiling points and the higher the saturated vapor pressures are, the faster the solvents evaporate. Viscosity is an indication of liquid mobility, which is strongly affected by the interactions between the molecules. For consolidation purposes, the lower the material viscosity, the better the material penetrates the substrates.

The mass ratio of menthol to solvent is 10. The solvent usage is as minimum as possible as mentioned earlier. For simplicity, all sol samples are named as M-solvent. For example, M-ethanol means menthol in ethanol (mass ratio of menthol/ethanol is 10). Moreover, M-melt means melted menthol. Viscosities of as-prepared sols at 20 °C and menthol melt between 45 and 90 °C were measured on an Anton Paar (MCR 102) Modular compact rheometer.

Table 1 Some basic physical properties of the solvents applied

	Methanol	Ethanol	1-propanol	2-propanol	*n*-hexane	Cyclohexane
Viscosity @20 °C(mPa·S)	0.544	1.074	2.256	2.4	0.307	0.94
Boiling point (°C)	64.7	78.5	97.2	82.5	68.7	80.7
Vapor pressure @20 °C (kPa)	12.97	5.870	1.941	4.418	16.16	10.34
polarity	5.1	4.3	3.9	3.9	0.1	0.2

Evaluation of menthol sols as temporary consolidants

Shore hardness was used to follow the solidification of menthol sols and melt. Certain amount of menthol sols or 50 °C melt were poured into PP molds (Φ = 50 mm, h = 10 mm) at 20 °C. The molds sat in air at ambient conditions for menthol sols or melt to solidify. The Shore hardness were measured on a l×-D Shore hardness tester over time.

The penetration depths of menthol sols and melt were measured as following, and the process was also illustrated briefly in Fig. 1. Four mockup samples were packed tightly in glass tubes (Φ = 20 mm, h = 90 mm). 5 mL of menthol sol or melt at 50 °C was added into these tubes dropwise until they were all completely solidified after sitting at 20 °C after 48 h (based upon Shore hardness test). The depths were measured using a ruler. Then, the samples in the tube were taken out. Unsolidified part of sample flew away and the solidified part was cut at positions of 3 and 6 cm respectively (see Fig. 1c). Depending on the consolidation results, 2 or 3 cut columns were obtained. These columns were weighed (recorded as W0), then were put in an air circulating oven at 110 °C for 12 h to completely remove all menthol before they were weighed again (recorded as W1). The amount of menthol in each part was calculated as W0–W1. The distribution fraction in each tube was calculated accordingly. The results reported were averages from three parallel tests.

The consolidation ability was further evaluated by impact strength on sand, which was measured using an Instron 5449 Universal Tester as described below. Menthol sols (M-ethanol, M-1-propanol, M-2-propanol and M-cyclohexane) or 50 °C melt were added into 10 × 10 × 55 mm molds filled with Sand 60. The amount of menthol added was 1/3 of the sand mass. After complete solidification, the consolidated Sand60 samples were used in impact strength tests according Chinese National Standard GB/T 14,389 – 1993. The averages based on three parallel measurements were reported.

Kinetics of menthol removal via sublimation was investigated at 25 °C under forced ventilation (air flow speed 1 m/s, relative humidity 60%) by simply weighing method. The weights of the samples were recorded daily for 15 days.

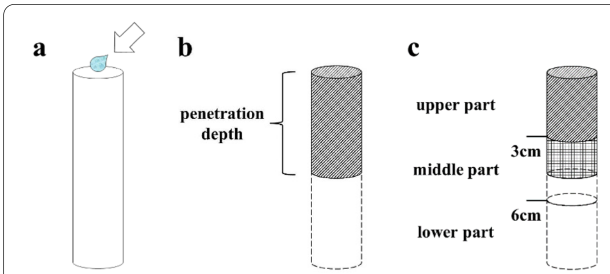

Fig. 1 Illustration of protocols for penetration depth measurement and menthol distribution determination

Result and discussion

Viscosity characterization of solvent-assisted menthol sols

Low viscosity is an important feature for consolidant molecules penetrating into heritages to achieve good consolidation outcomes. The viscosities of as-prepared menthol sols at 20 °C and menthol melt at 45–90 °C are shown in Fig. 2. It shows the menthol sols of ethanol, 1-propanol, 2-propanol and cyclohexane at 20 °C have viscosities comparable to that of a melt at 50–60 °C, which will benefit the penetration process. M-methanol sol has a viscosity around 38.6 mPa·s, which is much higher than other alcohol sols. It implies menthol molecules may have some strong interactions with methanol which has the highest polarity. As for *n*-hexane, the sol solidifies around 29 °C upon cooling. It appears menthol has lower solubility in *n*-hexane due to its nonpolar nature. The differences in viscosities of those sols indicate that sol viscosity is less relevant to the viscosity of the organic liquid used, but strongly affected by the polarity of the solvent applied. Based on viscosity data, M-methanol and M-hexane are excluded in the following studies.

Feasibility studies of solvent-assisted menthol sols as temporary consolidants

The penetration and consolidation experiments are performed on dried samples while the real condition at Liangzhu site is highly humid. This experiment protocol is taken because the water existing state in soil and sand are quite different so that it is difficult to achieve rationalized analyses and comparison of the data acquired on wet substrates.

Penetration behavior

The penetration depths of M-melt and four menthol sols in different substrates are summarized in Table 2. The data show that sols have much better penetration behavior than the melt. The melt shows similar shallow penetration depth on all matrices, suggesting that solidification upon cooling is the key to determine the penetration depth. As for the sols, data in Table 2 indicate that particle size of the matrix affects penetration strongly, consolidant sols penetrating much deeper in bigger particles. More detailed penetration information is revealed by Fig. 3. For substrate with small particles, the closer to the surface, the higher the menthol concentration. Menthol content decreases as the sol proceeds, which is consistent with observations on terracotta samples previously [14]. However, for substrates with larger particles, menthol distribution is the opposite, i.e., the closer to the

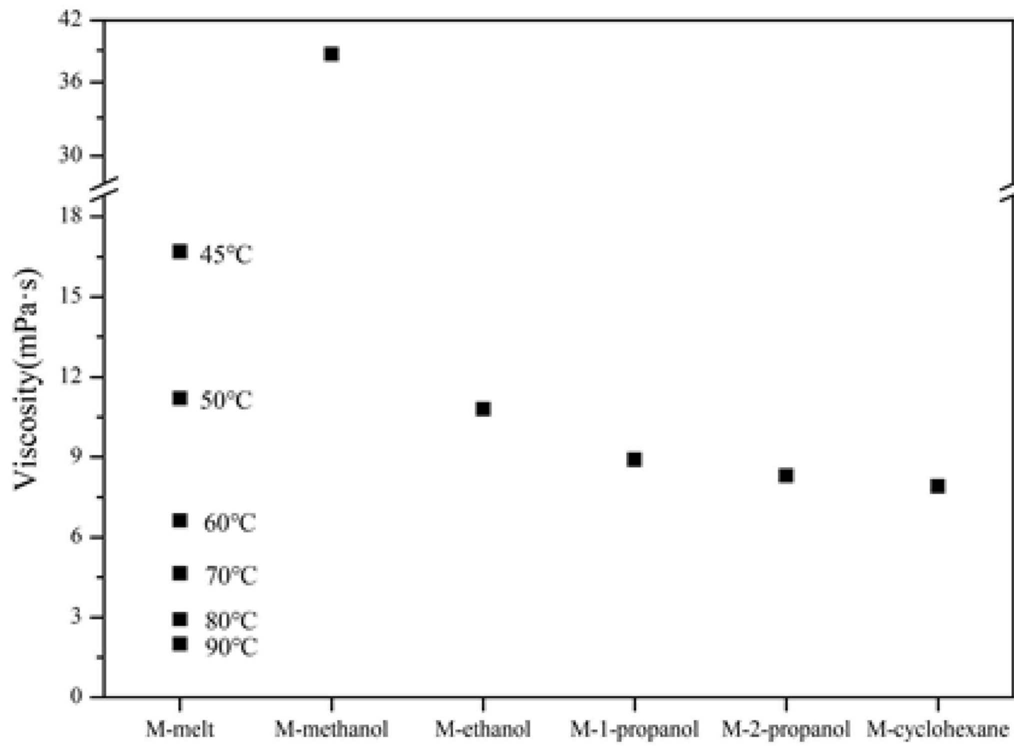

Fig. 2 Viscosities of solvent-assisted menthol sols @20 °C and menthol melt @ 45–90 °C

Table 2 Penetration depth (in cm) of solvent-assisted menthol sols in different substrates

	M-melt	M-ethanol	M-1-propanol	M-2-propanol	M-cyclohexane
Sand30 (30–40 mesh)	< 1.0	> 9.0	> 9.0	> 9.0	> 9.0
Sand60 (40–60 mesh)	< 1.0	6.5	4.7	5.4	4.7
Soil-LZ (40–60 mesh)	< 1.0	4.5	4.5	3.6	4.0
Kaolin (40–60 mesh)	< 1.0	5.1	4.8	5.0	3.8

surface, the lower the menthol concentration. It reminds us that special attention should be paid to ensure decent consolidation of the surface layer during applications, especially for highly porous items with large pores which are often fragile.

The penetration difference of sols on various substrates seems less dependent to sol viscosity but more dependent to the solvent polarity. The penetration depth is roughly smaller for less polar solvents. Apparently, menthol molecules have stronger interactions with more polar solvent molecules, thus can go further along with the solvent. This phenomenon is commonly observed in elution process in liquid chromatography [28].

Consolidation efficiency

Shore hardness was used to follow the solidification process of menthol melt and sols. Data in Fig. 4 show that menthol melt reaches its maximum hardness within one day [20]. Moreover, as expected it takes much longer for sols to reach their maximum hardness as solvent evaporation takes more time. The hardness from sol consolidation is slightly weaker than that from the melt. But they are all over 70HD, close to that of polystyrene, which we believe is decent for temporary consolidation of fragile heritages [29].

Impact strength tests are further carried out to evaluate the consolidation outcomes of menthol melt and sols when applying on mesh 60 standard sand samples. The data show that the mechanical strength of the sand samples consolidated by solvent-assisted menthol sols is slightly lower than the sample consolidated by melt. These impact strength values are comparable to paraffin (0.2–0.3 kJ/m^2)[30], which is good enough for temporary consolidation purpose. More detailed information

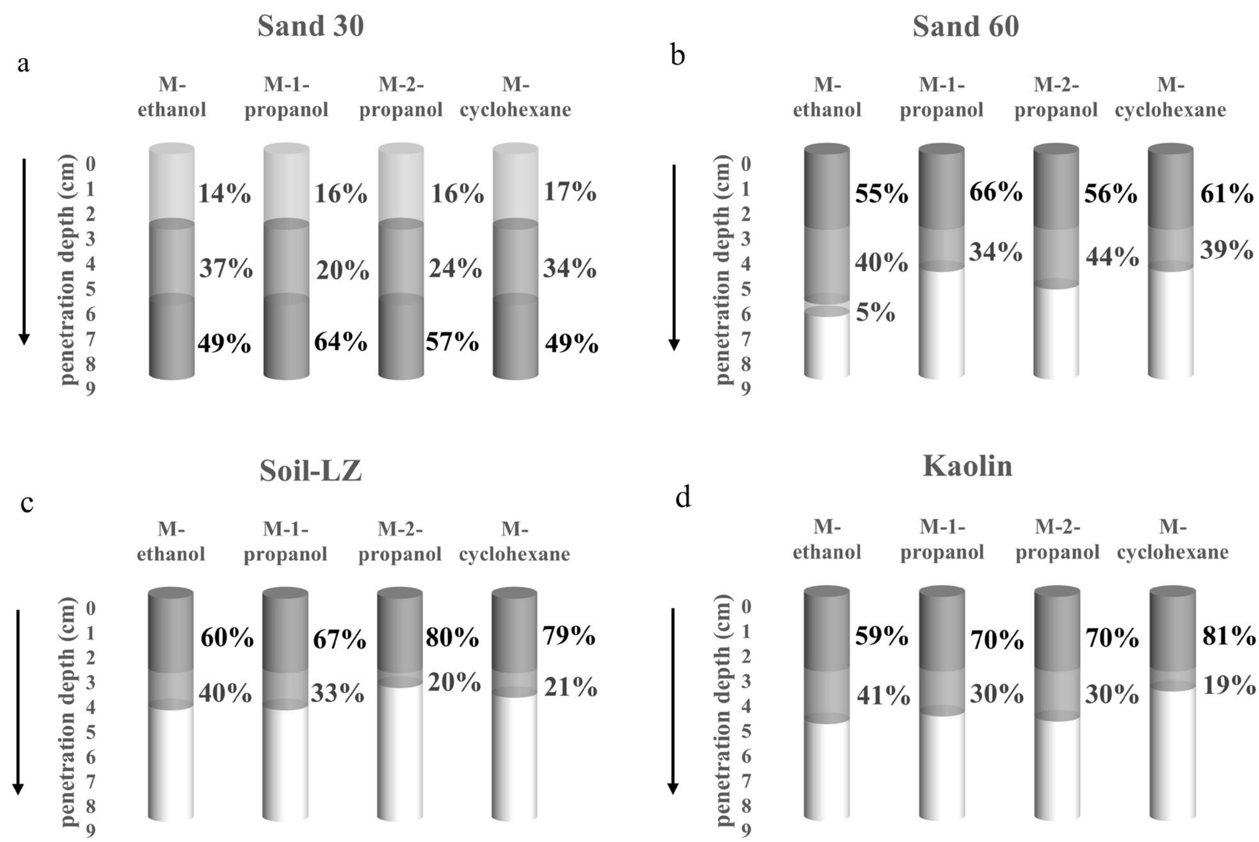

Fig. 3 Menthol distribution in different substrates in the sols. **a** sand 30, **b** sand 60, **c** soil-LZ, and **d** Kaolin

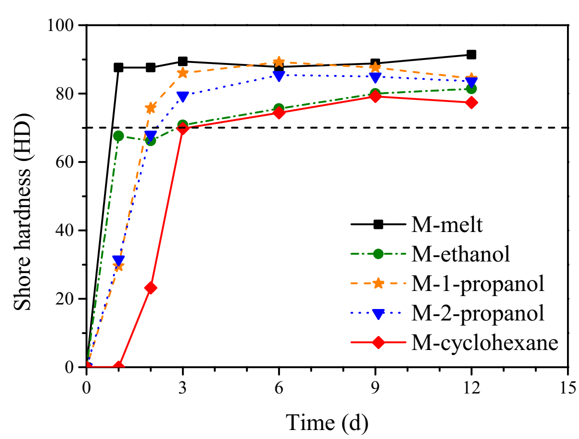

Fig. 4 Shore hardness of solvent-assisted menthol sols and menthol melt

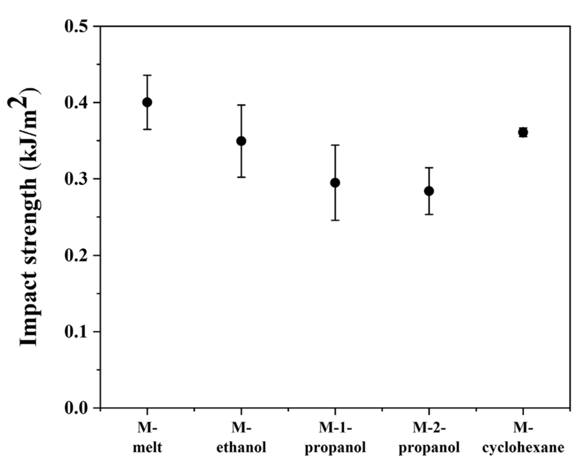

Fig. 5 Impact resistance of sands samples after being consolidated by solvent-assisted menthol sols and menthol melt

can be revealed by Fig. 5: the impact strength of sand sample consolidated by M-cyclohexane is closest to that of menthol melt while the impact strength of sand sample consolidated by M-2-propanol is the lowest. Interestingly, faster solvent evaporation (larger vapor pressure) leads to better consolidation strength but lower hardness.

Controllable removal of menthol after consolidation

Complete removal of consolidant is an important and mostly welcomed feature of temporary consolidation technique. The progresses of menthol removal via its sublimation on soil samples are shown in Fig. 6a. Data in Fig. 6a show that menthol can be totally removed after 12 days in all cases as expected [11,12,14]. The faster sublimation kinetics of menthol sols is probably due to residual solvent evaporation. The nonlinear sublimation kinetic patterns in all cases are consistent with previous observations [4, 14].

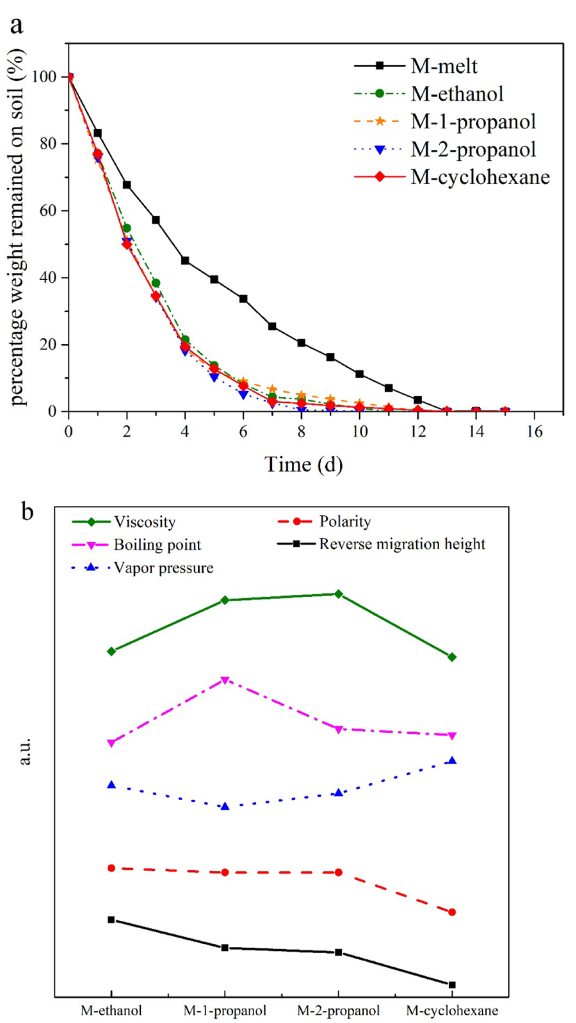

Fig. 6 **a** The sublimation processes of menthol sols and melt on soil samples; **b** reverse migration height of menthol of corresponding sols during removal with respect to the viscosity (20 °C, mPa·S), polarity, boiling point and vapor pressure (20 °C, kPa) of the solvents

During menthol removal, white menthol crystal whisker formation can be clearly seen on the soil surface for menthol sols, which is the most observable difference between menthol sols and the melt. During menthol removal, solvent evaporation can bring part of sols back to the soil surface, which is referred as "reverse migration". Once the solvent evaporates at the surface, menthol will crystallize to form whiskers. The whiskers form on the surface and grow towards the air, which is determined by its growth mechanism [31]. So, whisker formation will not cause any damage to the heritage, but it will reduce the consolidation strength since there will less consolidant inside the heritages. The height of whiskers formed on the soil surface of menthol sols, as an indication of degree of reverse migration, are compared in Fig. 6b. Apparently, reverse migration in M-cyclohexane sol is the lowest of the four sols, which also partially contributes to its highest consolidation strength. Comparing reverse migration heights to the solvent properties listed in Table 1 indicates that solvent polarity is the key factor here as well. Menthol molecules will have stronger interactions with more polar molecules. Thus, on one hand they can penetrate deeper with more polar solvent as discussed previously, on the other hand more menthol molecules will reversely migrate to the surface with more polar solvent evaporation. As shown and discussed in previous studies, temporary consolidation process has little impact on the samples physically or chemically32,33. The sols are safe to heritage relics.

Preliminary application of M-ethanol sol at Liangzhu ancient city

Liangzhu ancient city is an important Neolithic human cultural sites in China, which was listed as World Heritage in 2019. In preliminary application studies, M-melt and M-ethanol sol were used to consolidate soil samples saturated with water. As shown in Fig. 7a, the wet soil samples are from the ancient river course in Zhongjiagang at the Liangzhu ancient city, a predominant cultural deposit of the Liangzhu period. The soil samples reinforced with bamboo mat are saturated with water, as free water can be observed on its surface (Fig. 7b) [34].

Because it is difficult to determine the consolidant penetration depth or mechanical strength onsite, weight of soil lifted by the consolidant is used to evaluate consolidation performance instead. After removal of surface free water using dry cotton cloth, consolidation material is applied using Pasteur pipettes. 10 g of M-melt can consolidate and lift 4.1 g of wet soil due to its poor penetration and weak adhesion ability on wet sample. Moreover, 10 g of M-ethanol sol (containing 9 g of menthol) can consolidate and lift 14.0 g of wet soil, which is three times more than the melt. M-ethanol sol is selected mainly due

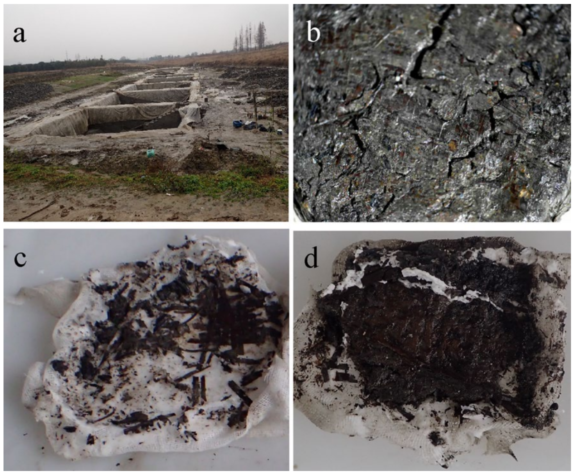

Fig. 7 **a** A picture of Zhongjiagang at the Liangzhu ancient city; **b** soil sample which is going to be consolidated and lifted; **c** consolidated then lifted sample using M-melt as consolidant; **d** consolidated then lifted sample using M-ethanol as consolidant

to ethanol low toxicity and easy accessibility. Although menthol and ethanol are considered to be low toxic, it is still recommended to wear suitable personal protection devices (masks, glasses, etc.) during application. The sol shows much better consolidation ability than the melt for water-saturated samples. Meanwhile, it is worth mentioning that lifting ability of cyclododecane, a well-known temporary consolidant widely used out of China, is zero due to its highly hydrophobic nature.

Figure 7c is the consolidation and lifting outcome when using M-melt as temporary consolidant, while Fig. 7d is the outcome using M-ethanol sol as temporary consolidant. In Fig. 7c, only part of the bamboo mat is saved. In Fig. 7d, the bamboo mat together with the wet soil is saved satisfactorily. Similar to results in the laboratory, M-ethanol sol shows good consolidation performance in wet condition onsite.

Conclusions

In this paper, a variety of solvent-assisted menthol sols are prepared, and the feasibility of these sols used as temporary consolidation material during archaeological excavations is systematically examined. The sols show much better penetration behavior than traditionally used menthol melt, which will greatly benefit consolidation applications under low environment temperatures or wet conditions. Good consolidation strength which is comparable to the melt can be achieved, while all chemicals used can be completely removed via evaporation and sublimation as the melt. Experimental data show that sol behaviors such as the penetration, sublimation etc. are mostly influenced by the solvent polarity, other properties such as boiling temperature, vapor pressure, viscosity and so on are less important. The hydroxy group in menthol molecules can have stronger intermolecular interactions with more polar solvent molecules, which will lead to higher viscosity, better consolidation strength, more significant reverse migration. Apparently, ethanol with medium polarity is a better choice than other organic solvents including methanol. Menthol-ethanol sol is applied in the consolidation and extract bamboo mat at Zhongjiagang at the Liangzhu ancient city with satisfying results. Overall, both the laboratory experiments and onsite application show that menthol-ethanol sol can be a suitable temporary consolidation material for archaeological excavation purpose especially in extremely wet condition.

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Author contributions
WZ: conducted most of the experiments and data analyses, wrote the initial draft of this manuscript; XW: implemented the viscosity and sublimation experiments and data analyses; XH, CM: conducted the on-site experiments at Liangzhu; XH: conceptualized the research, interpreted data and finalized the manuscript; HL: helped to design the experiment setup, revised the manuscript. All authors read and approved the final manuscript.

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Availability of data and materials
All data analyzed during this study are included in this published article. Raw data are available upon request.

Declarations

Competing interests
The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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