LABORATORY RESEARCH INTO THE USE OF MENTHOL AS A TEMPORARY CONSOLIDANT FOR CONSERVATION ON ARCHAEOLOGICAL EXCAVATIONS*

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Due to its ability to evaporate completely at room temperature, menthol has recently been proposed for use as a new kind of temporary consolidant at the excavation site of Qin Shihuang's Terracotta Army. Compared to cyclododecane (CDD), the most widely used temporary consolidant, menthol has the advantages of a longer working time, a well-established safety profile and easy local availability. In this paper, in order to better understand the temporary consolidation behaviour of menthol and to guide its optimal use in the field, laboratory experiments are carried out on ISO standard sand substrate in conditions mimicking the situation of an archaeological excavation. Basic and important knowledge about menthol, which is related to its effectiveness in temporary consolidation, such as penetration into the substrates, the solidification process and the degradation of the efficiency of consolidation over the sublimation time, are systematically studied. The results show that the penetration of menthol is notably affected by the substrate's temperature, particle size, particle-size distribution and water content. The solidification of melted menthol is an obvious volume shrinkage process, and the shrinkage extent is greatly affected by the application protocol. The consolidated samples experience a dramatic promotion in compressive strength, and as menthol volatilizes, the consolidated samples will eventually become loose and finally collapse.

KEYWORDS: MENTHOL, TEMPORARY CONSOLIDANT, CONSERVATION ON ARCHAEOLOGICAL EXCAVATIONS

INTRODUCTION

At archaeological excavation sites, numerous fragile objects or weak materials, such as delaminated polychromes, weathered lacquers or textiles, metal corrosion products, sculpture fragments and extremely fragile mural paintings, are discovered. In order to subsequently lift or move them from the field to a restoration room or any other location, a pre-consolidation process is often required.

Temporary consolidants are favoured by many archaeologists and conservators, due to the volatility of these substances, namely the so-called 'volatile binder' (Jägers 1999). These materials are able to evaporate completely at room temperature in an appropriate time, and thus no further treatment is needed to remove them. Among these substances, almost all the interest is devoted to cyclododecane (CDD), which was introduced to the conservation field in 1995 by Hangleiter (Hangleiter and Jägers 1995). A great deal of literature has reported the successful application

of CDD to protect fragile objects, such as the moulding of fossil specimens during transportation (Brown and Davidson 2010), the consolidation of low-fired ceramics during desalination (Muros and Hirx 2004), the stabilization of a dolomitic column base stone at the Metropolitan Museum of Art in New York City (Stein *et al*. 2000), the immobilization of hundreds of pieces of stone armour discovered at the archaeological site of Qin Shihuang's Terracotta Army (Xia *et al*. 2005) and the relocation of a Taoist real-body clay sculpture found at Matou Mountain in Shaanxi Province, China (Ma *et al*. 2011).

On the contrary, other volatile binders—for instance, camphene, tricyclene and menthol—and their potential as temporary consolidants are somehow overlooked because of the popularity of CDD (Rowe and Rozeik 2008). In our previous work, laboratory research and *in situ* trials in Pit No. 1 at the archaeological excavation site of Qin Shihuang's Terracotta Army, the use of menthol as an alternative temporary consolidant has been examined (Han *et al*. 2014). Very encouraging results have been achieved. However, several basic scientific questions still need to be answered, including the following. To what extent will the menthol penetrate into the substrates? How does the solidification of menthol behave inside the treated objects? How can we define the efficacy of temporary consolidation and how can we quantify it? What are the effects of the application protocols on the consolidation treatment?

In this work, systematic experiments are carried out to answer or partially answer the questions mentioned above. The real objects found at any archaeological excavation site are precious, and thus it is impossible to perform experimental studies on those objects. Normally, artificially simulated samples are used instead. However, the simulated samples are usually quite complex, which will complicate the experiments and make it difficult to straighten out the correlations between the data. Therefore, ISO standard sand was chosen on purpose as the substrate in our experiments. Except for the substrates, the experiments were designed and conducted to mimic the conditions in a real archaeological excavation site. Based on our laboratory studies, a better and deeper understanding of some of the basic aspects of the physicochemical behaviour of menthol as a temporary consolidant can be achieved, which is extremely helpful for directing the optimal use of menthol in the field.

<div align="center">EXPERIMENTAL</div>

Materials

(-)-Menthol (98%, melting point 42 °C) was purchased from Aladdin Co. and used as received. ISO standard sand (SiO_2, > 99.9%), manufactured according to ISO679/EN196-1 and characterized to be pure quartz by XRD, was obtained from the Xiamen ISO Standard Sand Co. Ltd (China).

Sample preparation and instrumentation

All experiments were performed on the standard sand. The sand was sieved through 30, 40, 60 and 100 mesh filters. The sand samples in the range 30–40 and 40–60 and under 100 mesh sieves were collected and named as sand 40, sand 60 and sand 100, respectively. All sand samples were cleaned with water and dried in an oven at 105 °C for 4 h before use. The physical properties of the sand were examined according to the protocol described in JTG E42-2005 (Ministry of Transport, China 2005). An electrothermal thermostatic oven (DH-250, Beijing, Keweiyongxing Instrument Co., Ltd, China) was used to dry the sand samples. Compression tests were performed

on a universal test machine (Shenzhen, Sansi Measurement Co. Ltd, China) with a load cell of 1 kN, and with each datum being the average of five individual specimens. Morphological change was investigated by means of a three-dimensional microscope (VHX-2000, KEYENCE Co. Ltd, China). Images were recorded using a Canon IXUS camera.

The evaluation of menthol as a temporary consolidant

Three main forms of volatile binders are applied: melt, saturated solution and spray from an aerosol or heated spray gum, which have been well summarized in the literature (Rowe and Rozeik 2008). In order to minimize the experimental variables, only melted menthol was applied in the current studies.

Penetration behaviour

Penetration behaviour on dried sand A glass tube (Ø = 30 mm, h = 200 mm), filled with 40 g of dried sand, was thermostated at 40 °C for 4 h. Then, 10 g of melted menthol (60 °C) was poured into the tube carefully. After adequate solidification (20 min), the tube was broken at the bottom by a hammer, unconsolidated sand was blown away, and finally a menthol-consolidated sand cylinder specimen was obtained. The penetration depth of the menthol was measured on this cylinder specimen using a ruler, and the datum reported was the average value of the maximum and minimum depths. Parallel experiments were done on three individual specimens with each sand sample. The specimens prepared from the penetration experiments were further cut into several fragments to investigate the penetration distribution within the sand samples. The length of each fragment was measured using a ruler, and the volume was studied by drainage. The weight percentage of menthol was calculated on the basis of the following formula:

$$\text{menthol content } (\%) = (w_0 - w_1) \times 100 / w_1, \qquad (1)$$

where w_0 is the original mass of the consolidated sand fragment, and w_1 is the mass of the sand after complete evaporation of the menthol. The menthol's length distribution was described, as the menthol content changes along the direction of penetration. A similar experimental process was applied on dried sand at 20 °C, but with the use of various amounts of melted menthol.

Penetration behaviour on wet sand Fifty grams of dried sand was wetted with water. The water contents were controlled to be 5%, 10%, 15% and 20% (by weight), respectively. A glass tube (Ø = 30 mm, *h* = 200 mm) filled with wet sand was sealed and thermostated at 20 °C or 40 °C, respectively, for 4 h. Then 3 g of melted menthol (60 °C) was poured into the tube. After complete solidification, the penetration depth of the wet sand was measured in the same manner as used on dried sand, except that the unconsolidated wet sand was washed away by running water.

The role of the application protocol

In practical treatments, menthol is usually applied on an object several times to achieve the best consolidation efficacy, and the number of duplicate treatments required depends on the degree of vulnerability of the object in question. In this paper, the influence of duplicate treatments on the final consolidation outcome is evaluated. Three casting procedures of melted menthol were applied, named as cast1, cast2 and cast3. For the cast1 procedure, the whole of 7 g of melted menthol (60 °C) was poured into an 8 mL mould (20 × 20 × 20 mm). For the cast2 procedure, 7 g of

melted menthol was poured into the same size mould in two steps. The latter half amount of melted menthol was poured into the mould until the previous half had solidified completely. Similarly, for the cast3 procedure, three steps were applied. The experiments were conducted at two mould temperatures: $0\,°C$ and $20\,°C$, respectively (mimicking winter and early summer atmospheric environments at the excavation site of Qin Shihuang's Terracotta Army). After all the melted menthol had solidified, a certain amount of water was added to fill the vent formed at the top of the bulk samples. That amount of water was accurately measured to calculate the volume shrinkage of the menthol during solidification according to the following equation:

$$\text{volume shrinkage ratio } (\%) = (V \times 100)/8, \tag{2}$$

where V is the volume of filled water and '8' is the volume of the mould in mL. Each volume shrinkage ratio datum is the average of three individual specimens.

The compressive strengths of pure menthol were also measured on these bulks.

Sublimation of menthol in consolidated sand samples

Melted menthol ($60\,°C$) was poured into dried sand ($40\,°C$). After rapid mixing, the mixture was cast into a mould ($20 \times 20 \times 20\,mm$, $T = 20\,°C$), then cooled in a refrigerator ($T = -1\,°C$) for 1 h and then finally demoulded carefully. The obtained consolidated sand specimens were wrapped in Parafilm® to leave one surface open to the air for sublimation. The sublimation process was conducted by placing these specimens in an electrothermal thermostatic oven ($T = 35\,°C$, RH = 50%) for 1 month. Compressive strength tests were carried out at sublimation times of 0, 5, 12, 20 and 25 days, respectively. The tests were measured on a universal test machine, and the load–deflection behaviour can also be obtained from this test. Each point in the resulting plot stems from the average of measurements on five individual specimens.

Morphological observations

The superficial morphological changes of the consolidated sand during sublimation were investigated using a three-dimensional microscope. Images were captured at sublimation times of 0, 5, 12 and 20 days, respectively.

RESULTS AND DISCUSSION

The physical properties of standard sand

Table 1 shows the physical properties of ISO standard sand. As the sand particle size decreases, the accumulated density of the sand sample decreases, while the porosity increases. This is

Table 1   *The physical properties of ISO standard sand*

Sand sample	Particle size range (μm)	Accumulated density (g cm^{-3})	Porosity (%)
Sand 40	550–380	1.477 ± 0.001	44.05
Sand 60	380–250	1.430 ± 0.005	45.56
Sand 100	150–120	1.351 ± 0.000	48.66

because of the particle-size distribution of the sand samples. For example, for sample sand 40, its particle size ranges from 550 to 380 µm, the size span of which is much larger than other two, which means that more smaller particles can fill the space amongst larger ones, leading to lower porosity and higher accumulated density.

Penetration behaviour

As shown in Table 2, at 40 °C, menthol penetrates deeper in sand 40 than in the other two samples. Apparently, larger particles pack to form larger pores, which aid the transportation of menthol inside the sample. The penetration distribution of the menthol within the sand samples is studied and shown in Figure 1. This reveals that the menthol content increases with the depth of penetration in the sand 40 and sand 60 samples, whereas in sand 100, the menthol content is approximately constant and close to the saturation uptake (approximately 26%). It is obvious that menthol is distributed more homogeneously in sand 100 due to its narrower particle-size distribution; and, as an outcome of that, consolidated sand 100 shows the highest compressive strength (Table 2). All these results suggest that the particle size and size distribution of the material to

Table 2 *The penetration depths of melted menthol on dried sand at 40 °C and the compressive strength of consolidated sand samples*

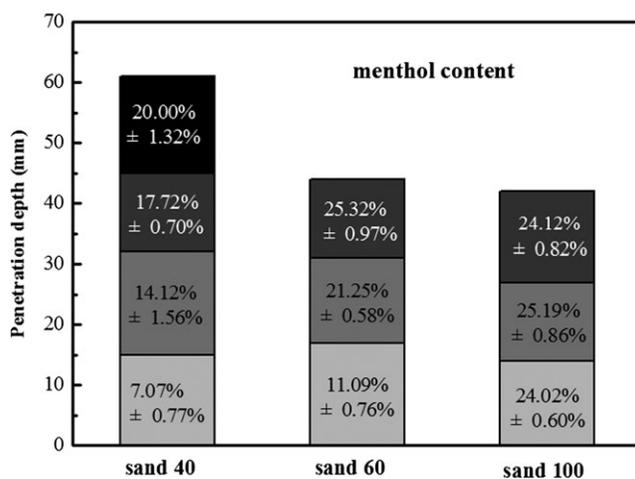| Sample	Penetration depth (mm)	Compressive strength (MPa)
Sand 40	59.92 ± 2.72	2.50 ± 0.18
Sand 60	54.88 ± 3.27	3.49 ± 0.23
Sand 100	47.84 ± 1.55	4.52 ± 0.31

Figure 1 *The penetration distribution of menthol applied on dried sand at 40 °C along the penetration direction: (a) sand 40 with menthol contents of 7.07%, 14.12%, 17.72% and 20.00%, respectively; (b) sand 60 with menthol contents of 11.09%, 21.25% and 25.32%, respectively; (c) sand 100 with menthol contents of 24.02%, 25.19% and 24.12%, respectively.*

which the menthol is applied greatly affect the penetration behaviour of the menthol and the mechanical strength of the object after consolidation.

Penetration depth tests on dried sand at temperatures lower than 40 °C were also conducted, and the results are quite interesting. Apparently, when the sand temperature is much lower than the melting point of menthol (42 °C), melted menthol will solidify significantly on the substrate surface, and the greater this degree of undercooling, the faster the menthol will solidify. The solidified menthol will hinder further penetration of the melted menthol. The penetration depth becomes independent of the quantity of menthol applied as illustrated in Figure 2, which indicates that the degree of undercooling becomes the overwhelming factor for the penetration behaviour of the menthol. Unlike the trend at 40°C, at 20 °C melted menthol is transported a shorter distance in sand 40 than in sand 60 and sand 100. This is probably because sand 40 has a slightly larger accumulated density (see Table 1). Melted menthol solidifies faster in sand 40 since more heat can be taken up by the same volume of sand. Again, the degree of undercooling is the key factor here. One thing worthy of further note is that an insufficient penetration depth for temporary consolidation is risky and our studies show that simply applying more consolidant will not solve this problem.

Comparing the penetration depths of sand 40, sand 60 and sand 100, it can be concluded that the influence of the particle size on menthol penetration can be suppressed by temperature. Due to the limited penetration depth of menthol at 20 °C, no satisfactory samples can be prepared to yield valid compressive strength data.

Water, or humidity, is an important variable at an archaeological excavation site; thus penetration studies have also been conducted on wet sand, to understand the effect exerted by water.

The penetration depth data collected from sand samples with water contents of 5%, 10%, 15%, and 20%, at 40 °C and 20 °C, are summarized in Figure 3. It is very clear that water hinders the penetration of menthol in wet sand samples despite the particle size and substrate temperature. When the water content reaches 20%, the penetration depths in all samples are very small and close to each other. It appears that at high water content, water becomes the predominant parameter that affects the penetration of menthol. The influence of water can be attributed to two
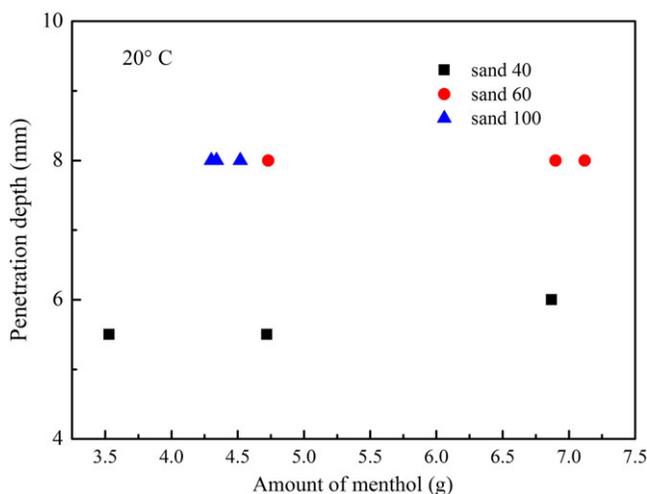

Figure 2   *The penetration depths of melted menthol on dried sand at 20 °C with various amounts of menthol. [Colour figure can be viewed at wileyonlinelibrary.com]*
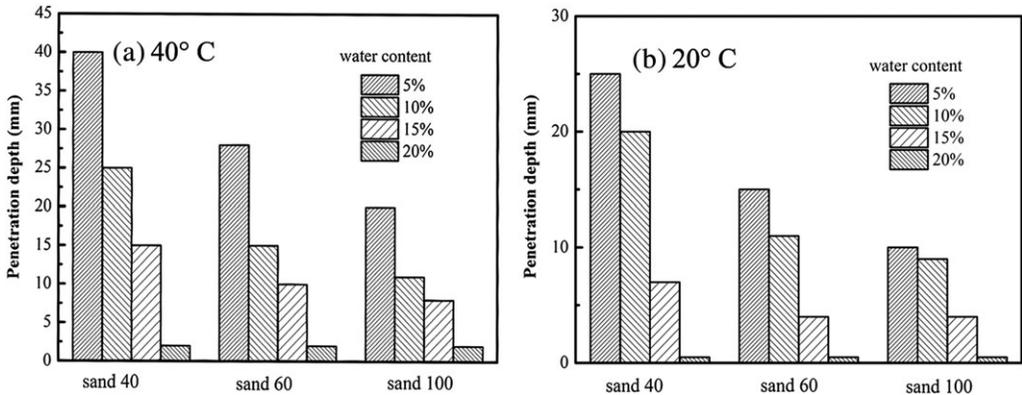
Figure 3 *The penetration depths of melted menthol on wet sand with varying water contents (5%, 10%, 15%, and 20%) and temperatures of (a) 40 °C and (b) 20 °C.*

causes: the competition for space between water and melted menthol and the hydrophobic nature of menthol. For wet sand, because of the incompatibility between water and menthol, the consolidation efficacy is very poor. Thus, no compressive strength results for wet sand have been acquired in this paper.

Very surprisingly, at 20 °C, the sand samples with lower water contents (5% and 10%) show greater penetration depths than dried sand at the same temperature (Fig. 2). It appears that the presence of water hinders the solidification/crystallization of the melted menthol, so that the melt can penetrate further. The water probably affects nucleation by changing the surface interaction between the melt and the sand. However, at high water content (15% and 20%), water will cause more undercooling and bring about increased competition for space, so that water hinders the penetration of menthol. In fact, once the water content reaches saturation value (approximate 26%), the melted menthol is floating on the surface of the sand, without any penetration (the density of menthol is less than that of water). This is an interesting and rather important finding for art conservation applications, since archaeological artefacts usually contain a certain amount of water. The underlying mechanism requires more systematic research, which is still in progress.

The role of the application protocol

As shown in Figure 4, the solidification of menthol begins from the melt/mould interface, and then grows solid towards the centre. As liquid is transformed completely into solid phase, a dent is formed in the centre of the bulk. Menthol obviously shrinks during solidification, which should be taken into account seriously, especially when it is being applied on fragile archaeological objects.

The menthol samples obtained under the various application protocols are presented in Figure 5. These samples are all white and waxy, with a clear translucent boundary line generated on the casting interface. Considerable reduction in volume shrinkage is found in cast3, especially compared with cast1. Specimens obtained from same mould under two different temperatures of application (0 °C and 20 °C) are also analysed in this test, and the results for the volume shrinkage ratios are shown in Figure 6. The three times casting procedure produces the smallest volume shrinkage, whereas for each casting method, a high mould temperature leads to a smaller volume change.

Such behaviour can be attributed to the degree of undercooling. It is well known that a larger degree of undercooling will favour faster nucleation, leading to smaller and more homogeneous
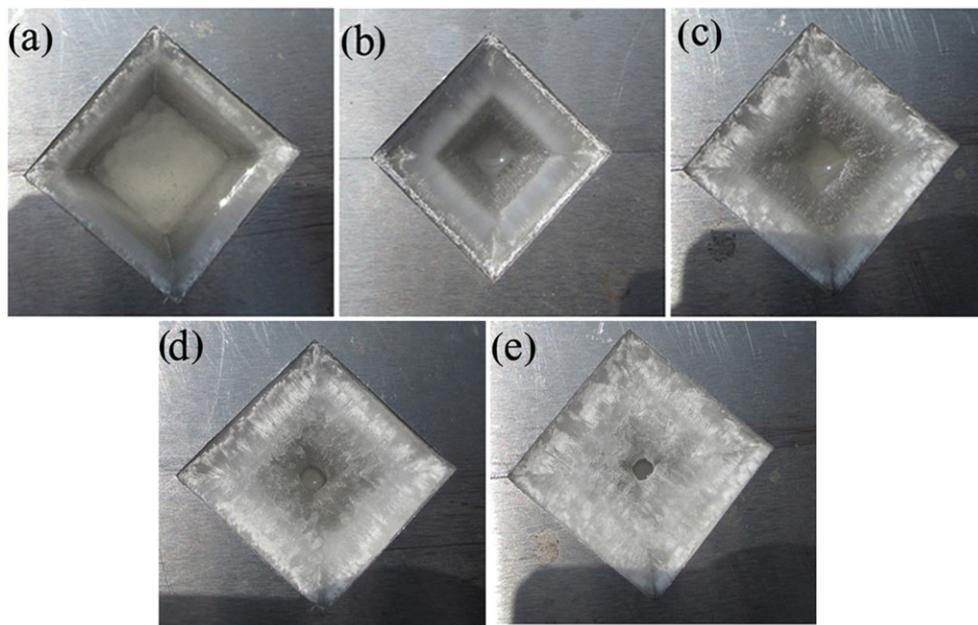
Figure 4   *Photographs of melted menthol during solidification, in chronological order. [Colour figure can be viewed at wileyonlinelibrary.com]*
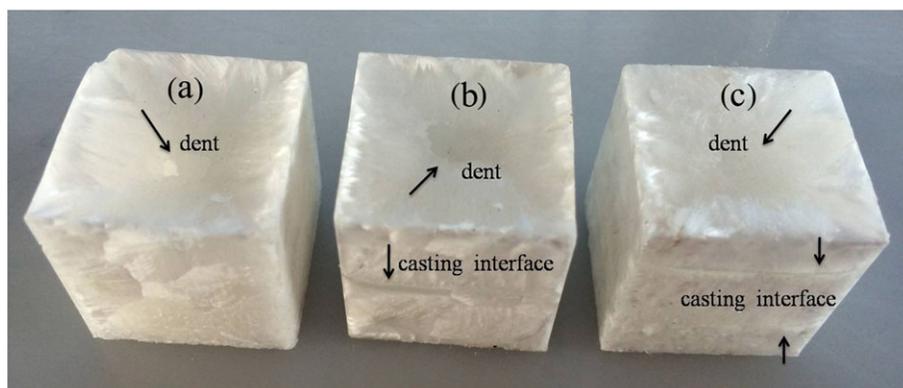

Figure 5   *Menthol bulks obtained from different application procedures: (a) cast1; (b) cast2; (c) cast3. [Colour figure can be viewed at wileyonlinelibrary.com]*

crystals (Hirth 1978; Graves and Perepezko 1986). In some extreme cases, an amorphous phase can be trapped by supercooling. For menthol, at 0 °C, faster nucleation and solidification lead to denser and more closely packed bulk materials, generating a smaller bulk volume (a larger volume shrinkage ratio) and better mechanical strength (Fig. 7). Smaller crystals provide a greater boundary surface to block slip or dislocation movements, thus also contributing to the higher mechanical strength. The results also reveal that duplicate treatment of melted menthol has the advantage of enhancing the mechanical strength and reducing the volume shrinkage. Such a phenomenon is not rare. In the polymer industry, it is common to use a multiple-step application
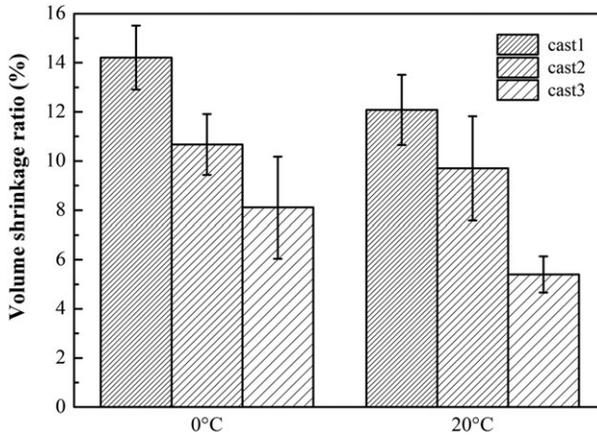
Figure 6   *The volume shrinkage ratio of menthol bulks obtained from the once-, twice- and thrice-cast application procedures, with the temperature of the mould at 0 °C and 20 °C.*
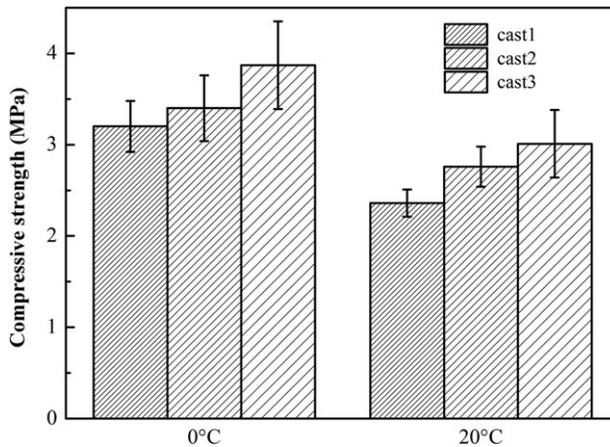

Figure 7   *The compressive strength of menthol bulks obtained from the once-, twice- and thrice-cast application procedures with the temperature of the mould at 0 °C and 20 °C.*

technique to reduce shrinkage during crystallization and to improve the mechanical properties (Nahill *et al.* 2003). Based upon the results, a multiple-step application technique is highly recommended.

The efficacy of consolidation and its degradation with sublimation

Compressive strength tests were carried out to determine the consolidation ability of menthol, and the data for consolidated sand samples are shown in Figure 8(a). Apparently, the mechanical properties of the consolidated sand are due to the consolidation of menthol, and only due to that mechanism. The original sand can be considered to have zero compressive strength. After the application of menthol, the mechanical strength of the menthol-consolidated sand samples is improved dramatically, which shows that menthol is an excellent 'binder'.
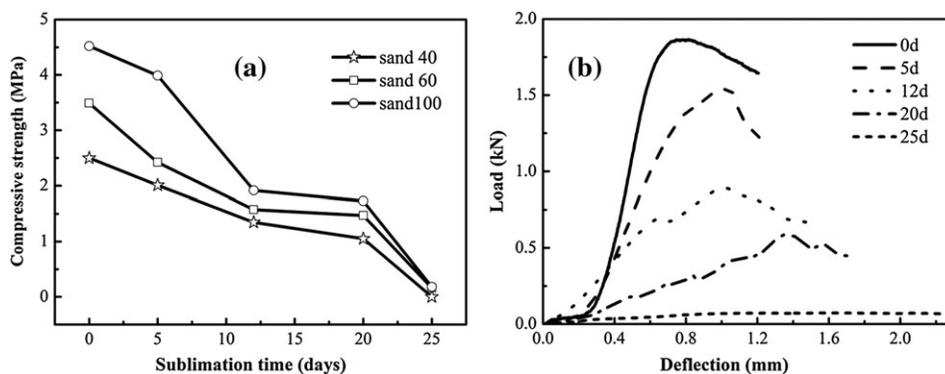
Figure 8   *(a) The compressive strength of consolidated sand 40, sand 60 and sand 100 in terms of the sublimation time. (b) Load–deflection curves of consolidated sand 100 in terms of the number of days of sublimation.*

The data in Figure 8 (a) further reveal that better consolidation results can be achieved for sand 100 (the compressive strength of consolidated sand 100 is almost twice as that of sand 40), which may be due to the stronger interfacial interaction in samples with smaller particles (it is known that the surface area increases as the particle size decreases) and more even distribution of menthol in sand 100, as demonstrated in Figure 1. Unfortunately, at the current stage, we could not determine whether particle size or size distribution was the more important consideration.

The term 'temporary consolidation' indicates that this protective treatment can only last for a short period of time, and that subsequent protective actions are required. The sublimation kinetics of volatile binders, including CDD and menthol, have been studied in laboratory conditions or in on-site applications (Chiara *et al*. 2011, 2014; Han *et al*. 2014). However, studies on the degradation of the efficacy of consolidation during sublimation, which is also very important, are very limited. In this study, the compressive strength of treated sand samples over the sublimation time has been examined. As expected, the mechanical strength of all kinds of consolidated sand samples decrease as sublimation proceeds, until all of the samples collapse completely (Fig. 8 (a)). The load–deflection curves for sand 100 over different sublimation days are shown in Figure 8 (b), which further demonstrates that the structure of the consolidated sand samples becomes loose and soft as the menthol volatilizes.

Direct observations of the degradation process of menthol-consolidated sand samples were acquired by means of a three-dimensional microscopy technique. The images are presented in Figure 9. The top view of freshly menthol-consolidated sand 40 is shown in Figure 9 (a) and its side view in Figure 9 (b). Obviously, the menthol functions as a 'glue' to make the sand particles stick together. As shown in Figures 9 (c) and 9 (d), menthol vanishes over time due to its sublimation. As displayed in Figure 9 (d), with the loss of the menthol 'glue', the superficial sand becomes loose and blows away, leading to exposure of the sand beneath and a continuous loss of menthol. Eventually, the once-consolidated sand samples will collapse completely. Such observations are in good agreement with the load–deflection curves obtained in the compressive strength tests.

CONCLUSIONS AND OUTLOOK

In this paper, we have presented the preliminary results of laboratory investigations related to menthol as a temporary consolidant for conservation on archaeological excavations. In order to simplify the experimental system, ISO standard sand was chosen as the substrate. The results
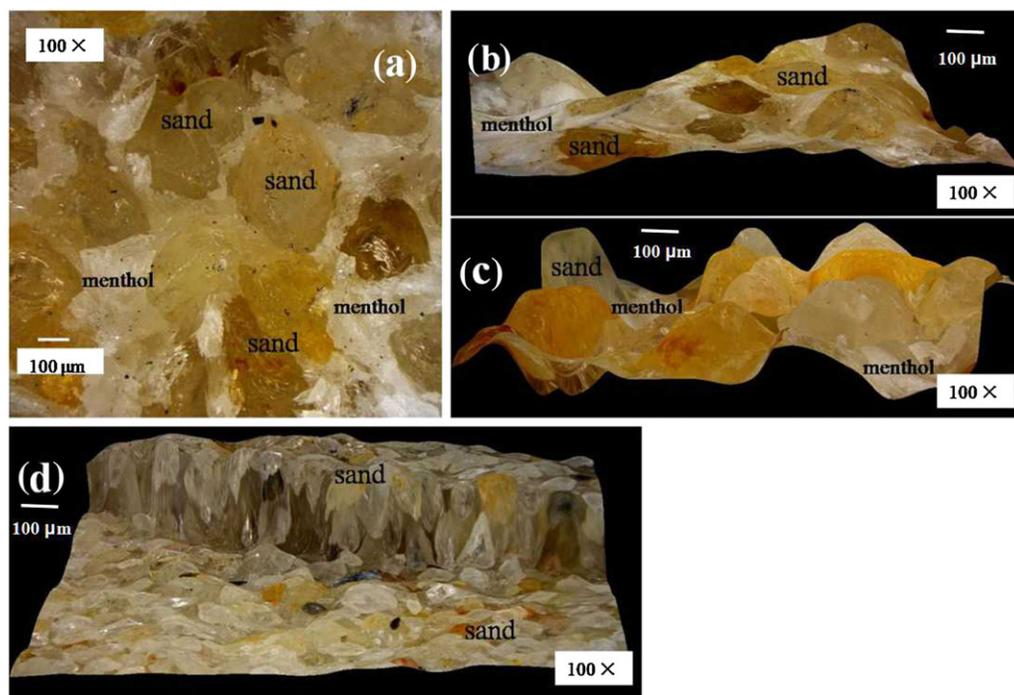
Figure 9   *Micrographs of menthol-consolidated sand: (a) original sand 40 (100×); (b) a three-dimensional image of (a) (100×); (c) sand 40 after 20 days' sublimation (100×); (d) sand 100 after 12 days' sublimation (100×), as menthol volatilizing the sand particles has become loose and led to exposure of the layer beneath. [Colour figure can be viewed at wileyonlinelibrary.com]*

show that the pore size and distribution of the substrate, basically determined by the constructed particle size and size distribution, have a strong influence on the penetration behaviour of the melted menthol. The temperature of the substrate also greatly affects the penetration behaviour of menthol, this being related to the degree of undercooling. Water will hinder the diffusion of melted menthol, and this can be attributed to competition for space and incompatibility between water and melted menthol. It is highlighted that the application protocol plays an important role, and that multiple repeated operations has the advantage of not only enhancing the mechanical strength but also reducing the volume shrinkage. Mechanical tests and three-dimensional microscopic observations show that as menthol volatilizes, the superficial sand of the consolidated sample becomes loose and subsequently blows away, leading to the exposure of the sand layer beneath. Such processes get repeated such that eventually the whole consolidated sample will collapse completely.

Overall, menthol is confirmed to be a promising temporary consolidant for conservation on archaeological excavations. The understanding of the basic physicochemical behaviour of menthol acquired in this study will help towards the optimal use of menthol in the field. Nevertheless, there are still many basic scientific problems that need to be solved. For example, objects excavated *in situ* usually consist of various materials, mostly in the form of extremely fragile organic matter, such as silk, lacquer, fabric, paper, wood and so on. These materials are always in an extremely perilous state, and some of them overlap, so the question of how to protect these artefacts remains a difficult challenge.

Further studies, aimed at protecting fragile organic materials using menthol, assessing its physicochemical behaviour on various kinds of matrices and evaluating its consolidation efficacy, both in the laboratory and in the field, are currently in progress.

REFERENCES

Brown, M., and Davidson, A., 2010, The use of cyclododecane to protect delicate fossils during transportation, *Journal of Vertebrate Paleontology*, **30**, 300–3.

Chiara, A., Federica, P., Brenda, D., Brunetto, B., Antonio, S., and Costanza, M., 2011, The study of cyclododecane as a temporary coating for marble by NMR profilometry and FTIR reflectance spectroscopies, *Applied Physics A*, **104**, 401–6.

Chiara, A., Federica, P., Brenda, D., Brunetto, B., Antonio, S., and Costanza, M., 2014, A non-invasive investigation of cyclododecane kinetics in porous matrices by near-infrared spectroscopy and NMR in-depth profilometry, *Journal of Cultural Heritage*, **16**(2), 151–8.

Graves, J. A., and Perepezko, J. H., 1986, Undercooling and crystallization behaviour of antimony droplets, *Journal of Material Science*, **21**, 4215–20.

Han, X., Rong, B., Huang, X., Zhou, T., Luo, H., and Wang, C., 2014, The use of menthol as temporary consolidant in Qin Shihuang's Terracotta Army excavation, *Archaeometry*, **56**, 1041–53.

Hangleiter, H. M., and Jägers, E., 1995, Flüchtige Bindemittel, *Zeitschrift für Kunsttechnologie und Konservierung*, **9**, 385–92.

Hirth, J. P., 1978, Nucleation, undercooling and homogeneous structures in rapidly solidified powders, *Metallurgical and Materials Transactions A*, **9**, 401–4.

Jägers, E., 1999, Cyclododecane, 14 February, http://cool.conservation-us.org/byform/mailing-lists/cdl/1999/0218.html (accessed 17 March 2015).

Ma, L., Zhou, W., and Qi, Y., 2011, Research on the relocation and conservation of the Taoist real-body clay sculpture at Matou Mountain in Shaanxi, *Sciences of Conservation and Archaeology*, **23**, 47–51. (in Chinese).

Ministry of Transport, China, 2005, *JTG E42-2005 Test methods of aggregate for highway engineering* (公路工程集料试验规程), 1 August.

Muros, V., and Hirx, J., 2004, The use of cyclododecane as a temporary barrier for water-sensitive ink on archaeological ceramics during desalination, *Journal of the American Institute for Conservation*, **43**, 75–89.

Nahill, T. E., Barker, K. J., and Lynch, B. A., 2003, *Graded crystallization of container finishes*, US patent 2003/0194518 A1.

Rowe, S., and Rozeik, C., 2008, The uses of cyclododecane in conservation, *Studies in Conservation*, **54**, 17–31.

Stein, R., Kimmel, J., Marincola, M., and Klemm, F., 2000, Observations on cyclododecane as a temporary consolidant for stone, *Journal of the American Institute for Conservation*, **39**, 355–69.

Xia, Y., Weichert, M., Zhang, Z., Zhou, T., and Ma, S., 2005, Application of cyclododecane on one stone armour set excavation and restoration, *Sciences of Conservation and Archaeology*, **17**, 31–5. (in Chinese).