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Menthol-Based Extraction of Fragile Wooden Coffin Lid (7–10th Centuries CE) in Laboratory Archaeology Excavation

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Abstract: Block lifting is a key step in stabilizing and removing fragile remains at archaeological excavation sites. Due to its favorable working properties and adhesive effect, menthol has recently been proposed as a volatile binding medium for temporary consolidation in archaeological conservation. This paper presents a case study on the use of menthol in the extraction and restoration of a large wooden coffin lid, approximately 1.9 m long and 0.9 m wide, from tomb 11 (M11) at Xie'ertala, located east of a Xie'ertala town in Hailar City, Inner Mongolia, dating to the 7th to 10th centuries CE. This coffin lid had fragmented into numerous wooden pieces, and was preserved in a relatively arid steppe environment, necessitating the extraction of the lid as a consolidated block. The use of menthol for consolidating and lifting the highly fragmented wooden coffin lid was intended to preserve critical archaeological information while avoiding damage to the underlying objects. An analysis of the physicochemical properties of these wooden remains suggests that the timber used for the coffin lid belongs to a common pine species from the Hulunbuir region. The degradation of the coffin lid was relatively mild, as shown by Fourier Transform Infrared Spectroscopy (FT-IR) and Scanning Electron Microscope (SEM) results. Dynamic Vapor Sorption (DVS) tests indicated that the hygroscopicity of the archaeological wood was 23.4%, compared to 21.1% for the reference sample, demonstrating good environmental stability. The safety of menthol as a treatment for fragile wooden remains was evaluated by comparing changes in the morphological and porosity characteristics of the coffin lid before and after menthol treatment. After treatment, the widths of the fissures remained largely unchanged, with all relative variations being less than 1%, and the porosity as well as pore size distribution of the wood showed negligible changes. Gas Chromatography–Mass Spectrometry (GC-MS) results showed that only 0.6% of menthol residue remained after 8 days of sublimation. This pilot study demonstrates that menthol is a safe temporary consolidant for block lifting and offers a promising alternative to the widely used cyclododecane. In conclusion, this research provided a new approach for conservators to safely lift similarly large and fragile wood remains during archaeological excavations.

Keywords: menthol; wooden coffin lid; archaeological excavation conservation; block lifting

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1. Introduction

Conservation professionals have been increasingly involved in archaeological excavations with the recognition of the significance of immediate stabilization of the finds, both chemically and physically [1,2]. One key procedure is block lifting of the artifacts, which consists of removing the artifacts together with surrounding soil to avoid potential mechanical and environmental damage to the fragile objects [3]. In situ consolidation is necessary to enhance the strength and cohesion of related components, especially for structurally complicated and fragile remains. This may take the form of external support such as plaster bandages, polyurethane foam, and strengthening frames as well as consolidant applied directly to the remains. The removability/retreatability of these applied materials, especially the consolidants, are therefore important factors affecting further investigation and conservation treatment.

In this regard, volatile binding media such as cyclododecane (CDD), which sublimates at room temperature and vanishes from the objects without further removal treatment, has been widely used by conservators as a temporary treatment agent since its introduction into the conservation field in the late 1990s [4–6]. So far, CDD has been the most popular volatile binding medium used as a versatile material by conservators working on paper, stone, wall paintings, polychrome pottery, and other archaeological objects [5,7]. However, due to the concern about the effects of CDD on human health and the environment, its challenging working properties, and changing market forces, conservators are beginning to reconsider other materials, some similar but less-known volatile binding media compounds such as menthol, camphene, tricyclene, and cyclododecanone [5,8–11].

Among these proposed materials, menthol has been the most systematically researched alternative to cyclododecane. Menthol is the main constituent of peppermint oil, usually extracted from the fresh leaves of corn mint or peppermint. Because of its unique flavoring and remarkable biological properties, menthol is widely used in pharmaceuticals, cosmetics, tobacco, and candies. For this reason, menthol industries have fully developed in many regions across the world [12]. As a consequence, menthol is easy to obtain at a fairly low cost. Similar to CDD, menthol sublimes at room temperature due to its high vapor pressure. However, unlike the very limited application of CDD, menthol is used in many areas where it is in contact with humans, and as a consequence, the safety and toxicology of menthol have received considerable attention. According to investigations by FDA and OTC, menthol is considered safe at certain ingestion dosages [13].

Concerning the use of menthol in heritage conservation, compared with CDD (melting point 60 °C), menthol has a lower melting point (40–42 °C), so its working property is better and much easier to brush when using the pure melt. Furthermore, due to the OH group of the menthol molecule, it forms weak hydrogen bonds with water-containing substrates, which gives it good penetration and adhesive ability in wet or humid environments [14,15]. The best operating temperature range for menthol is 20–40 °C higher than its melting points, namely 60–80 °C, which is friendly to temperature-sensitive artifacts, such as textile or lacquerware. In addition, within that temperature range, menthol has better consolidation performance [14]. The recommended operation protocol is a multiple-step application technique [16]. When menthol is applied to the objects, it functions as a consolidant to make the substrate particles stick together. After a certain time, the added menthol will sublimate and leave no contamination on archaeological materials. The sublimation rate of menthol depends on climate and exposure conditions. Generally, it will become faster with the help of ventilation and heating. It is reported that after 70 days of sublimation on non-porous glass slides, no menthol can be found in the dichloromethane extracts of the sample by GC-MS analyses [14]. Preliminary application at the Qin Shihuang's terracotta army archaeological excavation site, in which one polychrome 'negative print' of a terracotta horse head and bronze arrow relics were successfully consolidated and transported from the excavation site to the laboratory, has been reported [14]. This research indicates that menthol has been systematically studied and shows promising potential for conservation application as an alternative to CDD.

The Xie'ertala cemetery, dated to the late Tang to Five Dynasties period (7th to 10th centuries CE), is located in a river valley known as Hailar, within the Hulunbuir grassland to the east of a Xie'ertala town, Hailar City of Inner Mongolia, China. This arid steppe area is considered to be of significance for the research of ancient Shiwei groups that were closely related to the origin of the Mongols [17,18]. Discovered in the 1980s, the Xie'ertala cemetery has undergone several excavation sessions in the past decade [19]. The depth of the Xie'ertala tombs was approximately 2 m. During the excavation in 2013, the coffin of the tomb labeled as M11 in Xie'ertala cemetery was block lifted along with its surrounding soil, mainly sand, from the archaeology site to the laboratory of the Hulunbuir Museum of Ethnology. It is meaningful to retrieve the coffin lid in one piece, which will not only provide excellent examples of the highly skilled woodworking of the Shiwei people but also offer great potential for the paleoenvironmental and dendrochronological research in this area [20]. However, the coffin had been buried in a relatively arid steppe area for approximately 1100 years, and the wooden coffin lid had cracked into numerous small pieces with many gaps and fissures. At the beginning of the excavation, the coffin lid was damp; however, it quickly dried out after being exposed. The wooden coffin was relocated to the Hulunbuir Museum, and the indoor laboratory archaeological work was conducted about one year later. The coffin was approximately 1.9 m long and 0.9 m wide, and there was soil/sand filling the spaces between the numerous wood fragments, as shown in Figure 1a,b. Over the course of 1100 years, the fluctuating water levels of the river led to alternating wet and dry conditions, causing pressure variations and corrosion, which contributed to the fragility and cracking of the coffin lid. It is very challenging to lift this large highly fragmentary wooden coffin lid in one piece without damaging the below. Considering the good consolidation performance and controllable sublimation ability of menthol, which have been systematically investigated previously [14–16], the use of menthol as a temporary consolidant for lifting the entire wooden coffin lid was proposed.

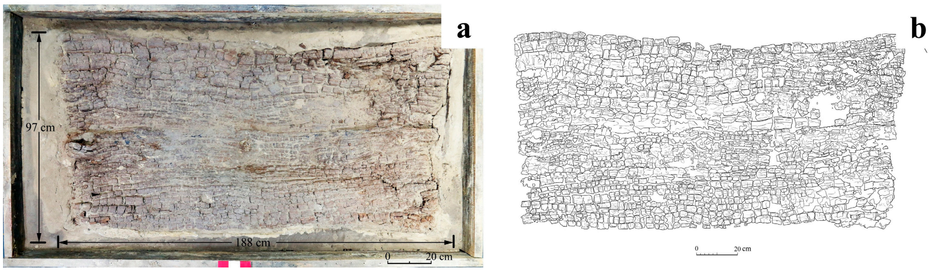

Figure 1. Photograph of the outer coffin lid of Xie'ertala cemetery M11 showing the serious degradation and fractured condition (**a**) and the drawing of coffin lid (**b**).

In this paper, menthol is used to consolidate and lift the large and highly fragmented wooden coffin lid of a tomb from the Xie'ertala cemetery during the indoor archaeological excavation. The physical effects of menthol's application on deteriorated wood and its applicability as a temporary consolidant for wood remains were investigated. The purpose of this report is to present a successful case study of menthol application on fragile wood remains for block lifting and to provide a reference for conservators when menthol is used in a similar situation.

2. Materials and Methods

2.1. Materials

The coffin lid samples (XT01, XT02, XT03) were taken from the outer coffin lid of Xie'ertala M11. M11 is a medium-sized rectangular tomb of a low-class Shiwei nobleman. The burial structure consists of inner and outer coffins with the outer one sealed with a ca. 1.9 m long and 0.9 m wide wooden coffin lid. The coffin lid was quite dry, and the preservation condition was good. The raw material could still be identified as wood, and the woodworking on the lid was still visible.

Menthol (99.8%, melting point 42 °C) was purchased from Aladdin Co. (Shanghai, China) and used as received.

2.2. Physicochemical Properties of Coffin Lid

Tree species identification: A small piece of wood from the coffin lid was sampled and identified at the Research Institute of Wood Industry, Chinese Academy of Forestry.

The cross, radial, and tangential sections of the sample were observed and compared with major wood species in China in accordance with (GB/T16734-1997) [21].

Assessment of degradation degree: Fourier transform infrared spectrometer (Nicolet iS 5, Thermo Fisher, Washington, MA, USA) was employed to analyze the degree of degradation of the coffin lid samples. The sound wood of *Pinus* sp. (Longyan City, Fujian Province, 25° N, 115° E, 390 m a.s.l., 35 years old, 30 cm DBH) was selected as the reference sample. The archaeological wood and reference wood were ground, mixed with KBr powder through further grinding, and then placed into a press mold for compression for 1 min. The results were obtained using an Fourier transform infrared spectrometer (FT-IR) within the 500–4000 cm^{-1} range, with a resolution using 16 scans. Scanning electron microscope (SEM) images were acquired using an ultra-high-resolution field-emission scanning electron microscope (Regulus 8100, HITACHI, Tokyo, Japan). Gold was sputtered onto the surfaces of samples as a conductive coating. The microscope was operated with an accelerating voltage of 15 kV, a resolution of 0.7 nm, a working distance of 15 mm, and a secondary electron imaging (Secondary Electrons) working mode.

Environmental stability: The moisture content under different relative humidity (RH) conditions was measured using a high-throughput dynamic moisture-adsorption tester (SPSx-1μ, ProUmid, Ulm, Germany). Preceding the test, the samples were air-dried at 103 °C for more than 24 h until their weights no longer decreased. The humidity range of this test was from 0% to 95% RH, changed in steps of 10% RH and a stable temperature of 25 °C. One adsorption and desorption cycle was measured. The default measurement frequency was every 10 min. The time taken for every step fell between a minimum of 60 min and a maximum of 600 min. The default weight limit had an upper cap of 1000%. The equilibrium condition was defined as $dm/dt \leq 0.01$.

2.3. Assessment of Menthol Extraction Performance

Micromorphological analysis: In order to assess the micromorphological changes in wood before and after menthol extraction, the ultra-depth-of-field three-dimensional video microscope (VHX-900, KEYENCE, Osaka, Japan) was employed.

Analysis of the pore structure: To study the impact of menthol extraction on changes in the pore structure of wood, the porosity analysis was characterized by automated mercury porosimeters (AutoPore IV 9500, Micromeritics, Atlanta, GA, USA). The surface tension of mercury was set as 0.485 N/m while the contact angle was set to 130°.

Residue analyses: After sublimation for 10 days, GC-MS was conducted to track any possible residue of menthol left in the wood sample. Approximately 1 g of the treated sample was immersed in 4 mL of methanol. The solution was shaken thoroughly and subjected to ultrasound for 30 min. The upper clear solution was collected and mixed with 2 mL of n-hexane, followed by allowing it to stand for phase separation, and the extract was collected from the n-hexane phase. This process was repeated three times. The combined extract was completely evaporated under a nitrogen stream, and then redissolved in 100 μL of n-hexane. An amount of 10 μL of a 1 mg/mL solution of n-hexatriacontane was added, followed by 25 μL of N,O-bis (trimethylsilyl) trifluoroacetamide (BSTFA) with 1% trimethylchlorosilane (TMCS, 99:1) for silylation. The mixture was heated at 70 °C for 1 h and analyzed by GC-MS for residual menthol. The residue of menthol was analyzed by a gas chromatography–mass spectrometer (GCMS-QP2020NX, Shimadzu, Kyoto, Japan), and the chromatographic column was an SH-Rxi-5Sil MS (30 m × 0.25 mm × 0.25 μm). The injection port temperature was set to 300 °C, using splitless mode with linear velocity flow control. The column flow rate was 2 mL/min, and the linear velocity was 51.3 cm/s. The initial temperature of the chromatographic column was 50 °C, held for 2 min, then ramped to 310 °C at a rate of 10 °C/min, maintaining this final temperature for 15 min. High-purity helium was used as the carrier gas, with an inlet pressure of 100 kPa. The electronic pressure control system operated in constant flow mode, and the mass spectrometer utilized electron ionization (EI) with an ionization energy of 70 eV. The mass-to-charge ratio (m/z) scan range was 50 to 800, with a scan cycle time of 0.3 s.

3. Results

3.1. Tree Species Identification

The cross (Figure 2a), tangential (Figure 2b), and radial (Figure 2c) sections of the sample from the coffin lid were observed and compared with major wood species in China (GB/T16734-1997) [21]. The sample had apparent growth rings, and the transition from earlywood to latewood changed rapidly. The longitudinal tracheid consists of a single bordered pit, the ray tracheid has dentate thickenings, and resin canals exist both axially and radially. These characters indicate that the wood is *Pinus* sp. of Pinaceae, which was one of the common species in the Gangga archaeological site in Hulunbuir steppe according to a report on ancient vegetation research [20].

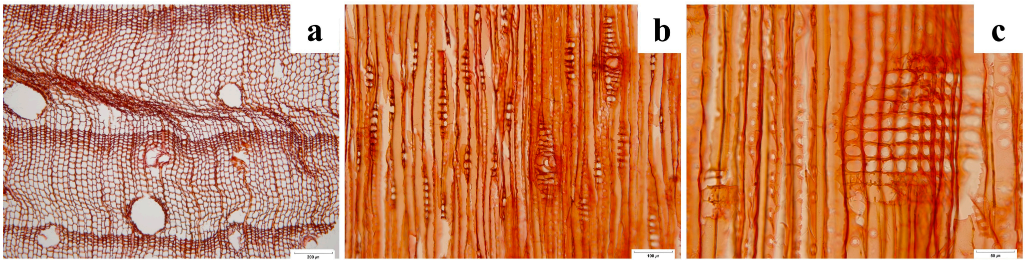

Figure 2. Anatomical structure of wood sample under optical microscope. The cross (**a**), tangential (**b**), and radial (**c**) sections.

3.2. Degradation Degree of the Coffin Lid

In comparison with the reference sample (Figure 3a), the FT-IR spectra confirmed that due to the degradation of cellulose, the absorption peak at 897 cm^{-1}, corresponding to the C-H deformation in the β-1,4 linkage of cellulose, disappeared [22]. Similarly, the peak at 1058 cm^{-1}, attributed to the C–O stretch in cellulose and hemicellulose, also vanished [23]. The peak at 1108 cm^{-1}, corresponding to the asymmetric stretching of C–O–C in cellulose and hemicelluloses, weakened in intensity in the archaeological wood [24]. Additionally, the peak at 1318 cm^{-1}, representing the CH_2 rocking vibration of crystalline cellulose, disappeared [25]. The band at 1372 cm^{-1}, which is due to CH bending in polysaccharides, became smoother in the archaeological wood [25]. Moreover, the peak at 1735 cm^{-1}, primarily attributed to the C=O stretch in acetyl groups of hemicellulose, also disappeared. This indicated that there is severe degradation of cellulose and hemicellulose in the archaeological samples, and the loss of polysaccharides suggests the possible oxidation of lignin. In general, despite the wooden coffin being buried underground for 1100 years, cellulose and other components underwent hydrolytic degradation. Additionally, over a year elapsed after the coffin was excavated before laboratory archaeological work and FT-IR testing were conducted, during which time lignin may have undergone further oxidative degradation.

Due to the relatively dry preservation condition of the coffin lid and the need to provide strategies for subsequent consolidation and protection, preliminary assessments of the wood's hygroscopicity, as well as environmental stability, were conducted using the DVS test. As the isothermal moisture adsorption curve of the sample (Figure 3b) exhibits an "S" shape, it indicates that both the archaeological and reference samples fall under the category of Type II moisture adsorption isotherms [26], suggesting that they have a high adsorption rate at initial humidity. As the relative humidity increases, the adsorption rate gradually slows down, yet they can still maintain a high moisture content at higher relative humidity. This suggests that the archaeological wood has barely mineralized and still retains the adsorption characteristics of cellulose materials. The equilibrium moisture content (EMC) of the archaeological wood is higher than that of the reference sample at any given relative humidity for the archaeological sample. Moreover, when the relative humidity exceeds 50%, the increase in the EMC rises more sharply with increasing humidity. At 95% RH, the EMC of the archaeological wood reaches 23.4%, compared to 21.1% for the reference sample. The factors affecting EMC include not only pore size and pore volume but also porosity. Compared to reference samples, the archaeological wood with severe degradation of cellulose and hemicellulose has more pores and a larger surface area. Since multilayer water adsorption is a form of physical adsorption, a significant portion of the adsorbed water theoretically consists of condensed water and capillary water within the pores. As the porosity increases, particularly the proportion of mesopores and macropores, the EMC is likely to increase accordingly.

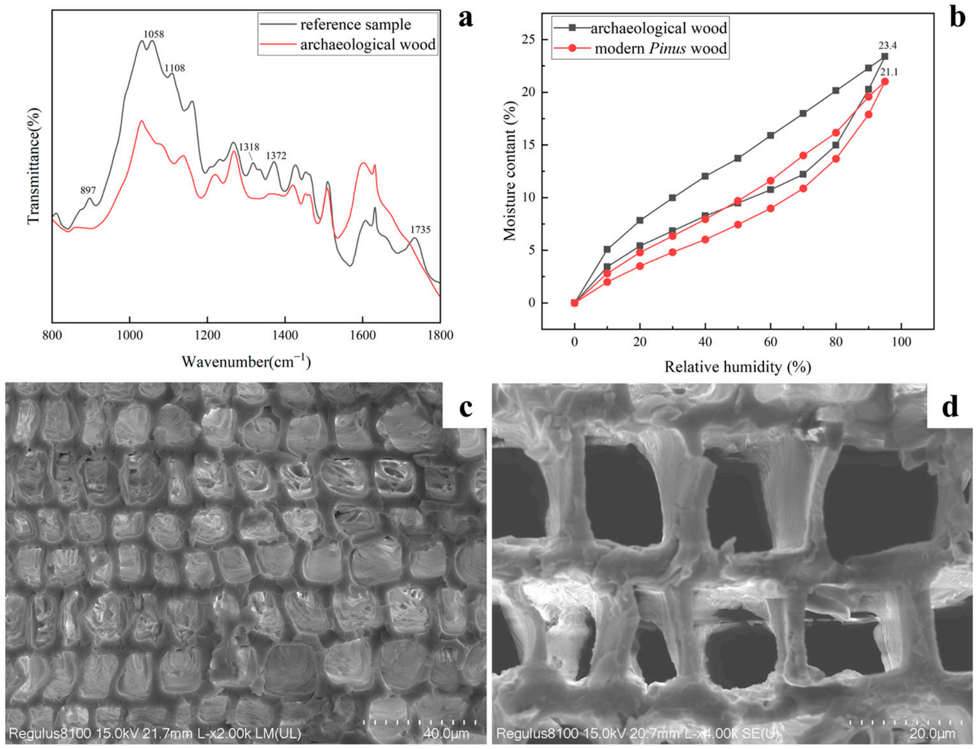

Figure 3. FT-IR spectra (**a**) and DVS curves (**b**) of the reference sample and archaeological wood; SEM of the archaeological wood ((**c**): 2000×, (**d**): 4000×).

In addition, the SEM result showed that the wood cell structure and cell wall of the archaeological wood were relatively complete, and no obvious shrinkage or collapse occurred (Figure 3c,d). FT-IR, DVS, and SEM indicate that the degradation of the wood samples is mild, but has good morphology.

3.3. Evaluation of the Extraction Performance of Menthol

Three small pieces of the wood fragment from the Xie'ertala M11 outer coffin lid were collected for the laboratory experiments. The surface morphologies of the original wood samples were carefully observed, the positions of existing fissures or cracks were marked, and their dimensions were measured (Figure 4a). Menthol was heated and melted in a water bath, keeping the melted liquid temperature at about 60 °C. The menthol melt was then dripped onto the wood samples using a Pasteur pipette. After about 2 min, menthol solidified completely and crystallized on the surface of the wood sample. To accelerate the menthol sublimation rate, an infrared lamp was used to heat the wood samples, during which the morphological changes of the wood sample surface were observed and recorded. The menthol-treated samples were then placed onto a glass plate without a lid in the laboratory at a room temperature of 25 °C and a relative humidity of 20%. After 10 days, no visible menthol crystals were observed on the wood samples (Figure 4d). The marked fissures and cracks were compared to the original ones, and the effect of menthol on the morphology of the wood samples was evaluated according to the changes in fissures or cracks' position and dimension. In addition, porosity and pore size distribution of treated wood were characterized by mercury intrusion porosimetry analysis (MIP), and the residue of menthol in the wood samples was tested using the GC-MS technique.

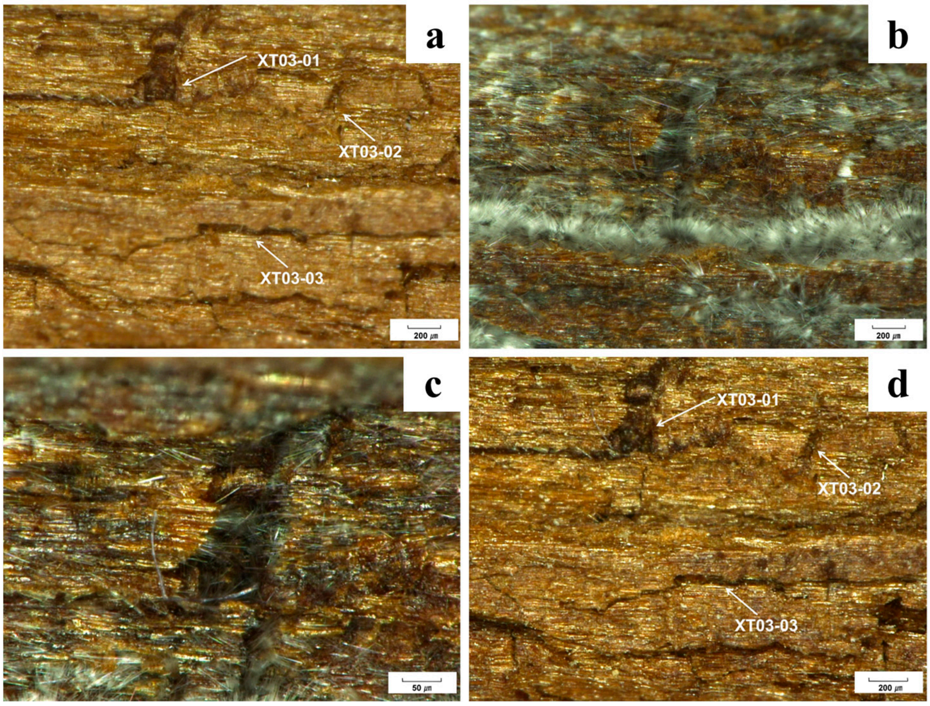

Figure 4. Micrographs of sample XT03 during the menthol treatment. (**a**): Untreated surface (50×), (**b**): 5 min after menthol treatment, newly grown menthol whiskers appeared (50×), (**c**): higher magnification of menthol crystals in wood fissures (200×), (**d**): 10 days after menthol treatment, no visible menthol was observed (50×).

3.3.1. Micromorphological Analysis of Menthol-Treated Samples

Figure 4 shows the surface morphology of the wood sample (XT03) at different stages during menthol treatment. When melted menthol flowed into the pores of the wood, it solidified quickly. As a consequence, the degraded wood fragments were consolidated. As menthol sublimated from the wood, obvious fine needle-like crystals started to form on the surface and within cracks of the sample (Figure 4b), which is consistent with the previous research [15]. Whether these whiskers led to any potential damage to the wood substrate was systematically assessed. The higher magnification image of menthol crystals in the fissure shows that these crystals grew toward open space, which would not damage the fragile wood (Figure 4c). After 10 days, no visible menthol was observed on the wood sample, but this does not indicate that the menthol had sublimated completely, because the pore structure of the substrate plays an important role in its sublimation behavior [14]. Therefore, more accurate methods like MIP and GC-MS were used to evaluate the residual menthol in wood samples and the effects of menthol treatment on the pore structure within the wood.

In addition, fissures of two other samples, XT01 and XT02, were observed and measured before and after the application of menthol. As shown in Table 1 and Figure 5d, the quantity, morphology, and position of cracks were almost identical to the original ones, and the widths of fissures remained substantially unchanged, with all the relative variations being less than 1%.

Table 1. Comparison of the fissure's widths in wood samples before and after the application of menthol.

Sample	Position	Crack Width (μm)		Relative Variation
		Original (w_0)	After Treatment (w_1)	$(w_1 - w_0)/w_0$
XT01	XT01-1	52.3	52.8	0.96%
	XT01-2	76.0	75.5	0.68%
XT02	XT02-1	92.1	91.3	0.84%
	XT02-2	94.9	94.4	0.57%
XT03	XT03-1	308.3	308.5	0.09%
	XT03-2	86.8	86.7	0.17%
	XT03-3	75.8	76.2	0.42%

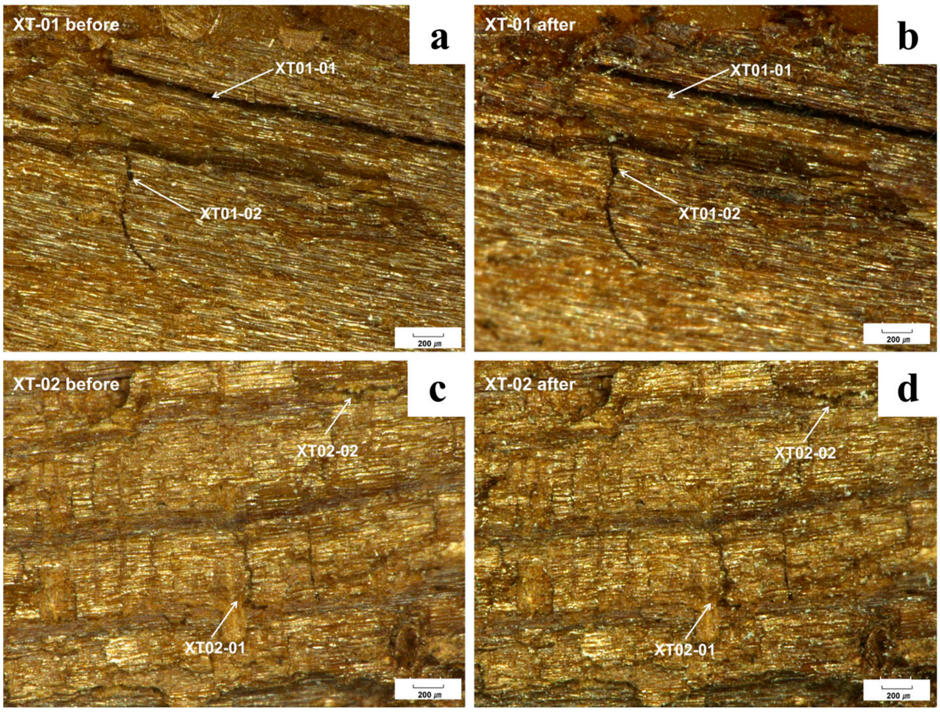

Figure 5. Micrographs of samples XT01 and XT02 showing the surface and fissures morphology before (**a**,**c**) and after (**b**,**d**) menthol treatment (50×).

3.3.2. Pore Structure and Residue Analysis After Menthol Treatment

Mercury intrusion porosimetry analysis (MIP) is a well-established method for determining the porosity and pore size distribution of materials [27,28]. By monitoring the variation of pore size, volume, distribution, and other porosity-related characteristics, one can evaluate changes in the structure and properties of porous materials. Figure 6a shows the pore distributions of both as-excavated (untreated) and menthol-treated (consolidated and then sublimated) samples from the coffin lid of Xie'ertala M11. The menthol-treated sample was measured after 10 days of sublimation. The untreated and treated samples used in MIP tests were sampled in the same location and have a similar early-to-latewood ratio. The trimodal pore size distribution of the treated sample remains basically the same as that of the as-excavated one. The untreated specimen has a porosity of 57.89%, while the porosity of menthol-treated specimens slightly decreased to 57.17%. This slight difference could be attributed to the measurement setup, as the MIP tests were conducted using different samples; therefore, the MIP data cannot be exactly the same. It is clear that menthol consolidation did not alter the porosity features of the tested sample. The MIP data are consistent with the morphology observations (Figure 5).

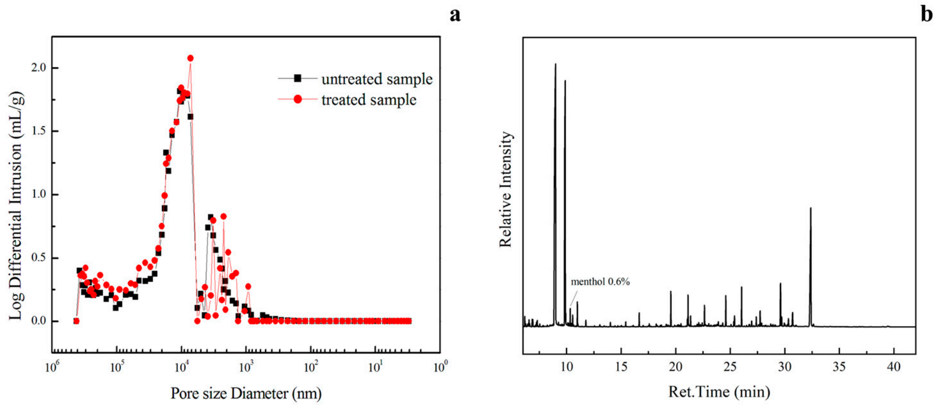

Figure 6. (**a**): Pore size distribution of wood samples (MIP); (**b**): residual amount of menthol after 10 days (GC-MS).

In order to further determine the residue of menthol in wood samples after volatilization, the samples treated with menthol were analyzed by GC-MS. As Figure 6b shows, only 0.6% of menthol remained in wood samples. This residue test was performed after 10 days of menthol evaporation. No menthol could be found after one month of sublimation on the coffin lid by GC-MS analyses.

3.4. Fieldwork Studies

Both morphological and MIP analytical results demonstrated that there was no notable alteration to the archaeological wood remains due to the usage of menthol for temporary consolidation. On the basis of this observation, a procedure for integrally lifting the coffin lid with temporary consolidation by menthol was conducted in the laboratory of the Hulunbuir Museum of Ethnology.

The treatment procedure is described in Figure 7. Before applying the menthol, the surface of the coffin lid was cleaned with a soft brush and rubber suction bulb to remove the dust and attached soil. Considering the large-scale, highly fragmentary wood coffin lid and its state of preservation, pre-consolidation was conducted before block lifting. In this case, the aqueous solution of peach gum (wt 37.5%) was chosen to strengthen the separated wood fragments following humidification of the surface by ethanol spray. As a kind of natural water-soluble resin, peach gum is often used to cohesively consolidate weak, highly fragile, or friable archaeological objects that have lost part or most of their original binders. After peach gum pre-consolidation, the coffin lid was then left indoors for air drying.

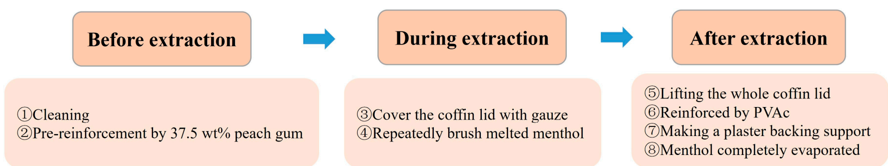

Figure 7. The procedure of using menthol as a temporary consolidant in coffin lid extraction.

After the coffin lid was air-dried, small pieces of cotton gauze were first overlaid in the areas where larger gaps were found. Melted menthol (60–80 °C) was then brushed in these areas to fill the gaps, providing extra reinforcement to avoid further separation and displacement of the surrounding pieces. Large pieces of cotton gauze were then overlaid, and the first layer of menthol was brushed (Figure 8a). The overheated menthol melt (60–80 °C) will have better liquidity and can penetrate the wood faster and deeper [14]. After the first layer of menthol was cooled and solidified, the menthol was brushed again to fill the fissures and promote further penetration into the coffin lid (Figure 8b). As all the menthol cooled and solidified, a temporary consolidated structure of cotton gauze and fragments of coffin lid adhered together by menthol was then formed and ready to be lifted and extracted integrally from the coffin (Figure 8c).

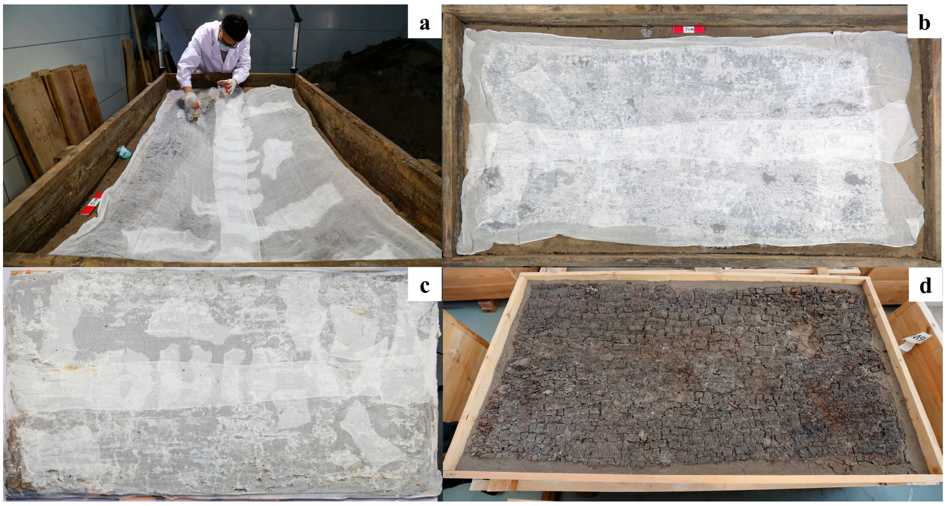

Figure 8. Photographs of the coffin lid in different stages of temporary consolidation and following treatment. (**a**): Brushing melted menthol; (**b**): finished temporary consolidation; (**c**): after 8 days of menthol evaporation; (**d**): display of the coffin lid after about one month.

In the following step, the back of the coffin lid was reinforced with PVAc and plaster, followed by supporting with a wooden frame. After 15 days, most of the applied menthol had sublimated, and the cotton gauze was ready to be removed. After further treatment on the front of the coffin lid, such as cleaning and filling the fissures, the coffin lid reached an integral and stable condition and was ready to be preserved in the storeroom or displayed in the exhibition hall (Figure 8d).

4. Discussion

In this case of using menthol to extract the large coffin lid from Xie'ertala, some valuable experience and techniques were gained. To optimize penetration depth and reinforcement strength, the temperature of the menthol melt should be maintained between 60 and 80 °C [14]. The application protocol is significant, as conducting multiple repeated operations not only increases mechanical strength but also reduces volume shrinkage [16]. Wrapping with gauze enables the entire coffin lid to be reinforced as a composite material once the menthol has solidified. Additionally, the gauze can help reduce excessive penetration of menthol into the wood's pores, thereby minimizing the difficulty of subsequent removal. Research indicates that it is preferable to pre-reinforce relatively dry or damp geoarchaeological remains with chemical agents in advance. Therefore, it is essential to pre-consolidate the wooden coffin lid with peach gum, as this can prevent the disintegration of the coffin lid after the menthol volatilizes. Additionally, a significant concern is whether these volatile binding materials have an influence on the radiocarbon dating of artifacts. Research on cyclododecane suggests that it does not impact dating data. However, the organic samples used in the test were treated with hexane, ethanol, and methanol, which are not typically included in the standard carbon-14 dating protocol [29]. In our latest study on the radiocarbon dating experiments for the bamboo mat excavated in southern China, dating back approximately 2200 years, we found that residual menthol persisted even after the conventional Acid–Base–Acid treatment. In contrast, cellulose extracted from the bamboo samples was free of menthol and produced radiocarbon ages consistent with those of bamboo samples that had not undergone menthol treatment. Therefore, we recommend collecting artifact samples that have not been contaminated by menthol for radiocarbon dating and other scientific analysis tests.

5. Conclusions

The Xie'ertala M11, located east of a Xie'ertala town, Hailar City of Inner Mongolia, contains valuable cultural relics that provide archaeological evidence of the activities of nomadic tribes associated with the Xie'ertala Culture on the Hulunbuir grasslands between the 7th and 10th centuries CE. Nevertheless, due to the dry burial environment and the collapse of the tombs, the central part of the M11 coffin lid sank and fragmented into numerous wooden pieces. Additionally, extracting and reassembling the fragments one by one would disrupt the original spatial relationships within the coffin lid. Therefore, the integral extraction using menthol allowed for the preservation of the fragile and fragmented coffin lid without interfering with subsequent excavation procedures. Identification showed that the coffin lid was *Pinus* sp., which is a kind of pine commonly found in the Hulunbuir region. The results of FT-IR and SEM analyses indicated that the coffin lid exhibited only slight degradation, which is consistent with the DVS tests demonstrating its good environmental stability.

Menthol's unique thermal and physical properties, such as its relatively low melting point, polar structure, solidification at room temperature, retention of original form after sublimation, and low cost, make it an ideal consolidant for the temporary stabilization of fragile artifacts during archaeological excavations. In this study, we evaluated the extraction performance of menthol and its effect on wood. We carried out micromorphological analysis and pore structure analysis, and measured residual menthol on the wood. MIP testing revealed that the use of menthol did not alter the porosity or pore size distribution in the archaeological wood. In addition, GC-MS analysis showed that only 0.6% of the menthol remained after 15 days of sublimation, and it had fully evaporated after one month. The pilot application during the indoor excavation of Xie'ertala M11 successfully consolidated and allowed us to lift the outer coffin lid without breaking apart.

It is suggested that, in comparison with CDD, menthol-based material would be a feasible choice for the temporary consolidation/stabilization and for block lifting fragile wood remains, especially for in situ conservation during the archaeological excavation. Needless to say, the application of menthol should be carefully and systematically investigated before it can be applied in different cases, given the complexity of archaeological remains and their burial environments.

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