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PINE PITCH: NEW TREATMENT PROTOCOLS FOR A BRITTLE AND CRUMBLY CONSERVATION PROBLEM

NANCY ODEGAARD, MARILEN POOL, CHRISTINA BISULCA, BRUNELLA SANTARELLI, MADELEINE NEIMAN, GINA WATKINSON

ABSTRACT

In 2011, the Arizona State Museum was awarded a Save America's Treasures grant to carry out a conservation survey of more than 22,000 basketry objects, including over 4000 ethnological baskets. This survey identified around 100 pitch-coated baskets, and a significant number were found to be in extremely unstable condition. Severe oxidization of the pitch had resulted in cracked, crizzled, brittle, and crumbly surfaces. To prevent further damage, the baskets were triaged; those considered at imminent risk for loss were treated mid-survey using solvent reactivation. This interventive conservation treatment stabilized the remaining pitch surfaces, allowing the baskets to be safely moved to their new storage locations. It also enabled future analysis and the research of the materials and technology used to fabricate these unique baskets.

This project illustrates the value of combining survey, analysis, and treatment activities. It initiated a more in-depth analysis of the pitch used in Southwest Native basketry. Pitch samples from over 70 baskets were analyzed with Fourier transform infrared spectroscopy (FTIR). These results were compared to fresh pine resin heated to simulate traditional pitch processing. Differences in appearance among the pitch surfaces appeared to correspond to different processing regimes. FTIR analysis also confirmed that this conservation treatment did not alter the chemical integrity of the pitch coating.

1. INTRODUCTION

American Indians of the Southwest have long employed pine pitch as a surface coating for basketry. Various trees of the *Pinus* genus exude a thick resin that can be heated and applied hot as a coating to baskets to make them water tight (fig. 1).

An item-by-item conservation survey and storage upgrade project, funded with a national Save America's Treasures grant, included more than 22,000 objects with over 4000 ethnological baskets. Within the ethnology collections, 100 pitch-coated baskets were examined. Seventy of these baskets were determined to be highly unstable due to severe oxidation of the pitch coating. Surfaces were characteristically blanched, cracked, crizzled, brittle, and crumbly. Underpainted designs on many of the baskets had become obscured by oxidized pitch and soiling. Entire surfaces suffering active loss were often accompanied by small Ziploc bags of pitch crumbs inside the baskets, revealing an ongoing need for conservation.

2. OVERVIEW OF THE ASM PITCH BASKET COLLECTION

Pitch baskets were used for transporting water. Baskets may have been repaired over time for reuse but the damages from oxidation make this difficult to determine. Some scholars also indicate there may have been a spiritual function (Simpson 2003). As seen in figure 2, a majority of the pitch baskets in the ASM collections were made by Apache weavers, including the Western, San Carlos, White Mountain, Jicarilla, and Mescalero Apache tribes. Other tribes whose pitch baskets are in the collection include the Navajo, Northern and Southern Paiute, Havasupai, and Hualapai.

Fig. 1. San Carlos Apache woman carrying a pitch-coated water basket from a tumpline around her head (one typically sees use wear and breaks in basket tumplines, in the carrying straps and on the basket pitch coatings, which would have been in contact with the carrier's back) (Courtesy of A. F. Randall, ca. 1883, Western History/Genealogy Department, Denver Public Library)

Of the 100 baskets, 77% are twined, 20% are coiled, and 2% are wicker or twined technology, and the weaving materials are typically stems of Sumac (*Rhus trilobata*) and/or Willow (*Salix sp.*). Tanner (1982) has suggested that before the application of a pitch coating, baskets were often initially waterproofed with a caulking of juniper or other leaves rubbed into the interstices of the weave. While the application of hematite powder imparted a red tint, some baskets were also painted with commercially manufactured red paints. The color of the baskets is subjective, because of the variations in manufacture technology and additives. Ferg (1987) indicates that clan-related designs were brushed on with ground charcoal pigment and other designs were woven in with devils claw seed pod (*Proboscidea*

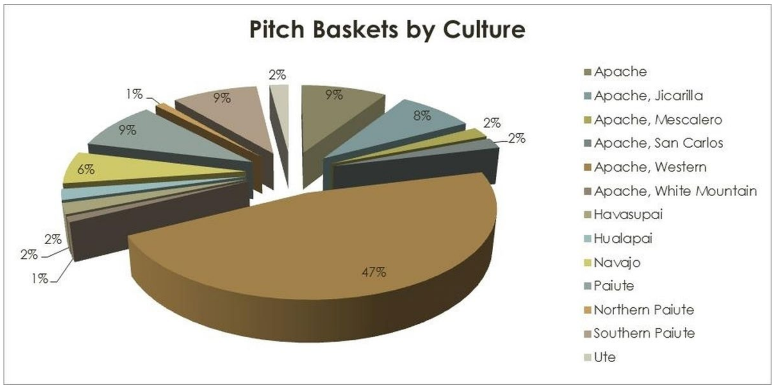

Fig. 2. Pitch baskets by culture at the Arizona State Museum

parviflora). Some Jicarilla baskets had pitch applied only to the interior surfaces with white clay applied to the exterior, and in some instances with polychrome-painted designs over the clay.

Among the possible sources of tree resins in the Southwest, the most common pines found at lower elevations are pinyon (piñon) pines (Richardson 2000) and many descriptions of pitch baskets specifically cite its use (Tanner 1982). Two species of highly resinous pines, *Pinus monophylla* and *Pinus edulis*, are also widespread (Fogg 1966; Richardson 2000; Smith 2000). Ponderosa pine (*Pinus ponderosa*) and Arizona cypress (*Cupressus arizonica*) are also resin producing trees in the area.

3. THE PROBLEM: OXIDATION AND ITS SYMPTOMS

It has long been observed that over time the pitch coatings on baskets oxidize because of environmental exposures including excesses of UV light, temperature, relative humidity, and pollutants in museum storage and exhibition that can cause a dramatic change in appearance. On the basis of empirical observations of the ASM pitch basketry collection, the more transparent and red pitch coatings tend to become discolored, crumbly, and brittle, resulting in flaking and the loss of surfaces (figs. 3–5). The darker, heavier pitch coatings tend to exhibit deeper cracking, discoloration, delamination, and have more localized loss with larger pieces rather than fine crumbles (fig. 6).

Fig. 3. Lighter-colored pitch exhibiting discolored, crumbling, and brittle surfaces due to oxidation (Courtesy of Marilen Pool)

Fig. 4. Cleaving pitch surfaces resulting in loss (Courtesy of Marilen Pool)

Fig. 5. Cracking and soiling of surfaces (Courtesy of Marilen Pool)

Fig. 6. Darker, opaque pitch exhibiting deep cracking, discoloration, and loss (Courtesy of Marilen Pool)

4. CONSERVATION TREATMENT PROTOCOLS

To prevent actively ongoing surface loss, these baskets were treated mid-survey. The intervention allowed baskets to be moved through the survey process and into their new storage location without additional damage and loss. This also assisted the curatorial activities related to how the baskets were placed in storage on the basis of their cultural designations and upgraded and improved the data entered into catalog records. The conservation treatments prompted further analysis and research on pitch basketry technology.

A literature review of pitch basketry conservation found no published articles; however, treatment reports and the evidence of treatments were noted in several museum collections. These include the use of B-72 acrylic consolidants, glycerin, and a butane torch. For example, a curator described his father's treatment of a pitch basket in the 1970s by saying, "When the torch was used, all the lumps were smoothed out and the appearance was glassy" (Ferg 2014). The results of these various treatments have not been particularly successful in stabilizing the pitch surface.

Conservators of paintings on canvas have previously noted the problem of aging pine resin coatings related to increasing oxidation over time:

The higher oxidized compounds formed in the *Pinaceae* resins during ageing are responsible, in some extent, of the changes in physical and chemical properties of the varnish film and, therefore, they affect directly the changes in solubility undergone by the varnish film. Thus, dissolution of higher oxidized compounds notably enhances the cleaning effect. The study carried out has proved that solvents exhibiting greater capacity for establishing hydrogen bond forces, such as ethanol, are the most effective for dissolving such compounds. Thereby, these solvents are especially effective for cleaning aged varnishes (Osete-Cortina and Doménech-Carbó 2005, 152).

Two considerations that guided the conservation treatment protocol were:

1. We did wish to consolidate the crumbs, integrate cracks, diminish scuffs and blanched areas, and restore some of the original intended gloss, but from a cultural perspective, we were hesitant to add new materials to the baskets. In conversation with Navajo weaver Etta Rock about the pitch basket appearance in 2004, conservator Dr. Nancy Odegaard noted that the look of a newly made pitch basket is always glossy. While it is understood that aged baskets will naturally become dull and deteriorate, Etta felt that weavers prefer to see them as they were made.
2. Selecting a method to reactivate the pitch surface without the use of a consolidant had been considered and tested on other materials by Odegaard and Pool (1999). On the basis of solubility testing and a desire to not introduce new and different material, the use of solvents to reactivate damaged coatings was considered an appropriate approach.

4.1 TREATMENT

To stabilize the damaged pine pitch surface, the baskets were first placed in a glove bag solvent chamber. A glove bag is a flexible, polyethylene chamber with built-in gloves that lets you work in a totally isolated and controlled environment. We placed the bag over a cube-shaped frame made of 1 in. PVC pipe fittings and sealed it with clamps.

An open container of ethanol was placed inside the glove bag for up to 24 hours for vapor conditioning of the surfaces (fig. 7). Sometimes dry surface particulates can be removed before reactivation if the surface is stable enough.

Fig. 7. Solvent chamber for conditioning pitch surfaces (Courtesy of Marilen Pool)

The baskets were moved to the fume hood and a fine mist of ethanol was applied with a handheld pressure sprayer to reactivate the damaged pitch and to stabilize the surface. Reactivated surfaces were initially tacky; therefore, the upper and lower portions of the basket were treated separately. After the first portion was reactivated, it was propped for drying on a plastic beaker to ensure the initially tacky surface did not adhere to anything else. Upon drying, the basket was inverted and the remaining untreated surface was reactivated. Brushes, Teflon film–covered swabs, and Kimwipe tissues saturated with ethanol were employed for cleaning and securing difficult surfaces. Handmade Teflon-covered swabs rolled across the pitch surface were especially useful for imparting a varied surface appearance during solvent reactivation depending on the individual basket (figs. 8, 9).

Fig. 8. Misting the damaged pitch surfaces with ethanol (Courtesy of Marilen Pool)

Fig. 9. During treatment, applying ethanol by brush (Courtesy of Marilen Pool)

An added benefit with this treatment approach is that the reactivated pitch can act to mend simple tears and breaks in the basket weave, eliminating the need for additional mending materials such as Japanese papers and paste. Even very structurally unstable baskets with large areas of loss were significantly strengthened and improved by stabilizing the surfaces. It should be noted that the baskets were allowed to dry for at least 2 days in our low RH controlled conservation lab before subsequent handling, documentation, and storage (figs. 10–12).

Fig. 10. Old storage for baskets at the Arizona State Museum (Courtesy of Nancy Odegaard)

Fig. 11. New storage for baskets at the Arizona State Museum (Courtesy of Marilen Pool)

Fig. 12. New storage for pitch baskets at the Arizona State Museum (Courtesy of Marilen Pool)

5. PINE PITCH ANALYSIS

During the treatment, we noted that not all pitch surfaces reacted the same to the application of solvent. Some pitch tended to solubilize more rapidly, reactivating and readhering to their woven surfaces with less pressure. In other cases, the pitch solubilized less rapidly, requiring more ethanol application between the pitch coating and basketry substrate as well as greater pressure to readhere loose pieces. The chemical analysis of detached and loose pine pitch particles addressed two questions: (1) Did the difference in solubility of pitch surfaces reflect different compositions? (2) Did the reactivation treatment chemically alter the pitch?

5.1 TREE RESIN COMPOSITION

Tree resins are terpenoid-based chemicals that are secreted at a site of physical wounding. They consist of higher-molecular-weight terpenoids with a lower-molecular-weight volatile fraction of mono and sesquiterpenes or phenolic compounds that act as a solvent to carry the larger terpenoids during secretion (Langenheim 2003). In most pine resins, the predominant resin acids are abietic and pimaric type terpenoids (fig. 13), but the components of pine resins will vary depending on species and other environmental factors (Joye and Lawrence 1967; Smith 2000; Silvestre and Gandini 2008). Fresh resin contains ~75% resin acids, with the remaining 25% being mostly volatile terpenoids and phenolics (Zavarin et al. 1971; Panda 2008). This fraction is terpentine and is primarily α- and β-pinene (Panda 2008). Pine species can show a large seasonal variation in components, particularly total phenolics (Nerg et al. 1994).

5.2 EXPERIMENTAL METHODS

Pitch from nearly 70 baskets in the collection were analyzed with FTIR to assess if there was chemical change as result of the treatment and to identify any compositional differences between pitch surfaces. In most cases, loose pitch fragments collected before conservation treatment were used. For comparison after treatment, small samples (< 0.5 mm^2) were removed with a scalpel. FTIR spectroscopy was performed on a Thermo-Nicolet iS10 spectrometer with a Smart-iTR attachment, equipped with a He-Ne laser and CCD detector. Spectra were recorded in reflection mode, from 4000 to 650 cm^{-1}, with 256 scans at 4 cm^{-1} resolution, and using OMNIC ESP 6.1a software.

Heating experiments were also carried out on raw pine resin to study changes in the chemistry of pine resins during processing. Samples of fresh resin were collected by removing excess exuded resin from the surface bark of pinyon pines from the Navajo Reservation in northern Arizona, approximately 30 miles west of Canyon de Chelly (probably *P. edulis* or *P. monophylla* based on geographic distribution). The samples were heated using a VWR 1320 oven to the temperatures described in section 5.3.2 later. The resin samples were then held at each temperature for 30 minutes and monitored with a thermocouple (Cole Parmer type J thermocouple). At this time, changes in flow properties and color were recorded. It should be noted that the collected resin contained contaminants, primarily bark, and other woody materials from the scraping of

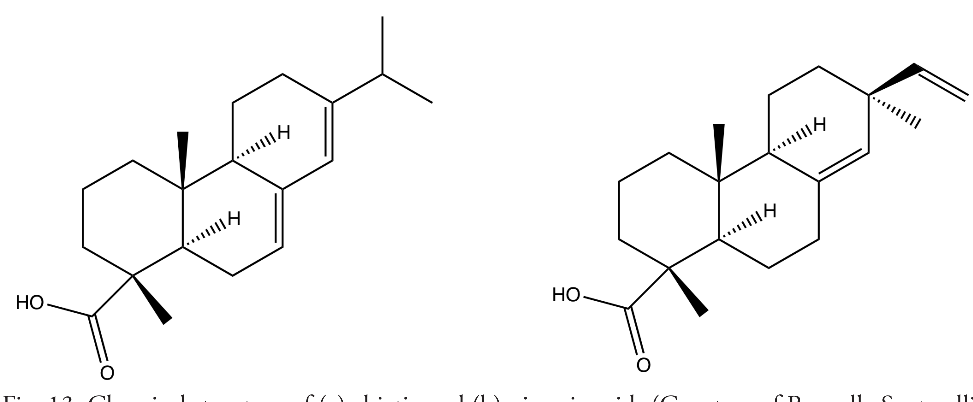

Fig. 13. Chemical structure of (a) abietic and (b) pimaric acids (Courtesy of Brunella Santarelli)

the resin from the trees. To identify the impact of these wood contaminants in these heating experiments, pitch samples were divided into two subgroups. In the first group, the resin was heated with the woody material present in it. In the second group, the resin was heated until fluid and the woody material was removed by pouring off the resin. FTIR spectra of the resin were collected after each heating step.

5.3 RESULTS

5.3.1 Analysis of Pitch from Baskets

Representative FTIR spectra of pitch samples removed from pitch baskets are shown in figure 14, and bands and band assignments are shown in table 1. Band assignments were made based on published studies of tree resins and processed pitch products (Robinson et al. 1987; Senftle and Larter 1988; Font et al. 2007). The spectra are consistent with diterpenoid resins and match reference spectra for pine resin

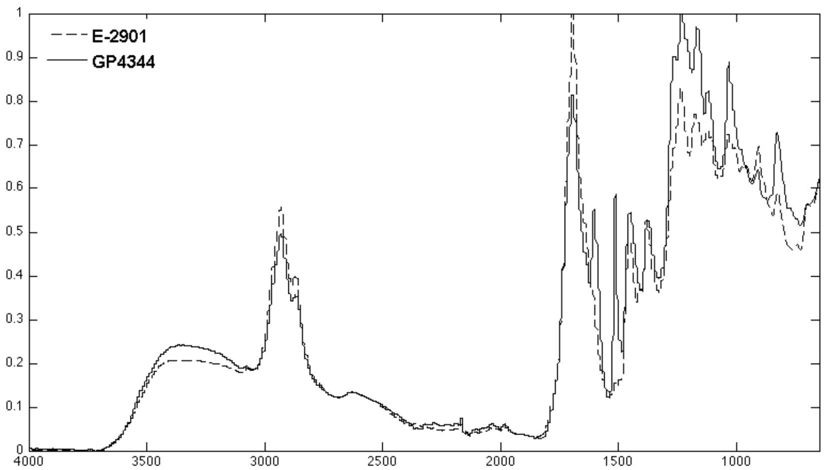

Fig. 14. FTIR spectra (absorbance units vs. wavenumber, normalized) of samples of pitch from baskets ASM E-2901 and ASM GP4344

Table 1. Band Frequency and Assignment of Typical FTIR Spectra of Pitch from Baskets (Courtesy of Christina Bisulca and Brunella Santarelli)

Band Frequency (cm)	**Band Assignment**
3070	(C=C)
2930–31	$\nu(CH_2)$
2869	$\nu(CH_2)$
1694	ν(C=OO)
1604–10	ν(C=C)
1514–16	ν(C=C)
1452–54	$\delta(CH_2)$, $\delta(CH_3)$
1375–83	$\delta(CH_3)$
1260–75	ν(C−O−H), carboxylic acid
1240–45	ν(C−O−H)
1123–26	ν(C−O−C), ester
1107–20	ν(C−O−C)
1032–35	ν(C−O)
908–10	$\delta(C{=}CH_2)$

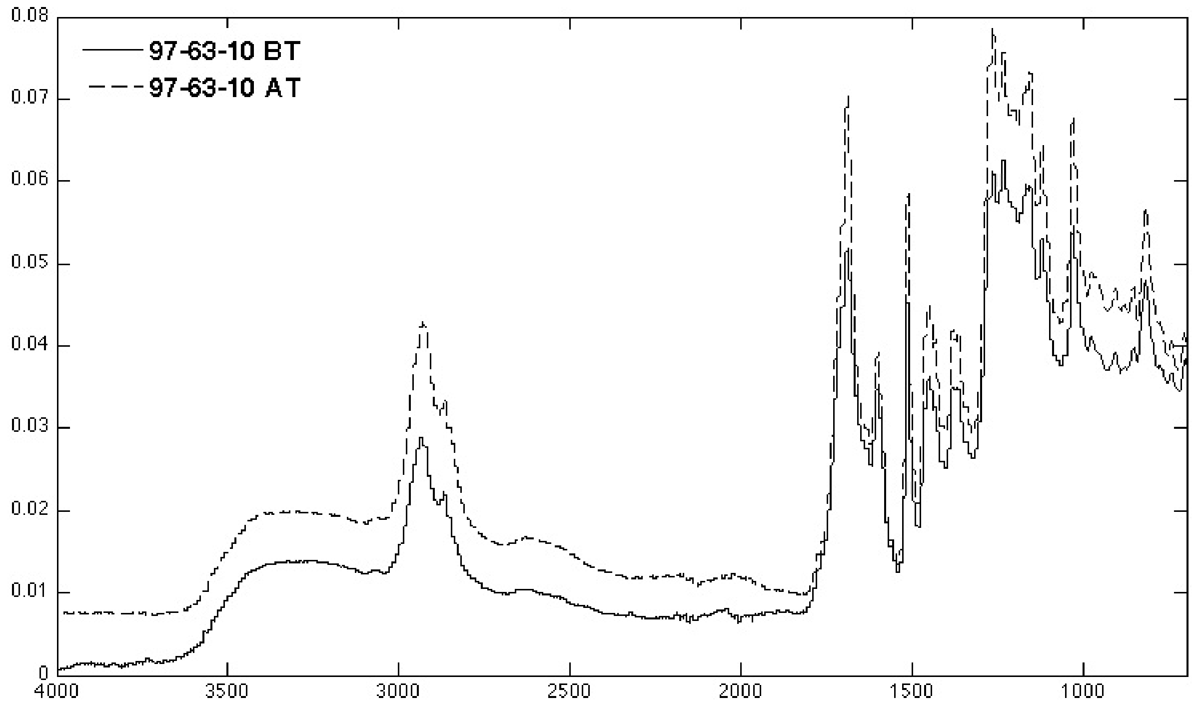

Fig. 15. FTIR spectra (absorbance units vs. wavenumber, normalized) of pitch sample before and after conservation treatment

(Derrick et al. 1999). It is, however, not possible to distinguish between various pine species with FTIR. The presence of abietic acid was also confirmed with the microchemical Raspail test that uses sugar and sulfuric acid to determine the presence of rosin (Odegaard et al. 2005).

Figure 15 shows the FTIR spectra from pitch before and after treatment with ethanol. No changes are observed after the conservation treatment, suggesting that this treatment does not interfere with the chemical composition of the pitch. Future analysis with other techniques such as gas chromatography-mass spectrometry (GC-MS) should provide more detailed information on the effects of solvent treatment.

FTIR spectra before treatment with ethanol revealed some notable differences in the pitch chemistry among the baskets sampled. Approximately 15% showed significant aromatic ν(C=C) bands at ~1605 and 1515 cm^{-1}, which are not observed in fresh pine resin. In other studies of pine pitch, these bands are attributed to aromatic groups formed during the processing into pitch (Robinson et al. 1987; Font et al. 2007). Figure 14 shows the FTIR spectra of pitch with and without an aromatic component. In general, the pitch coating of baskets with a significant aromatic component seen in the FTIR spectra is dark in color and opaque, similar to the pitch shown in figure 6. Those with a low aromatic component are typically translucent, and range in color from golden yellow, to red, to dark brown (figs. 3–5).

5.3.2 Heating Experiments

Heating experiments were also carried out using fresh pine resin collected locally. These experiments revealed that temperature directly impacted the color and working properties of the resin. The color changes observed during the heating of the resin are described in table 2. The fresh resin was a soft, opaque resin, white to yellow/brown in color (fig. 16). The resin became clear and translucent with low heating (80–90°C), and then hardened upon cooling (fig. 17). The resin did not become fluid enough to flow readily until it was heated to temperatures above 100°C. The resin remained a yellow color until a temperature over 180°C was reached, at which point the resin began to darken significantly (fig. 18). This color change appeared to be a function of high temperatures: resins heated at a constant 175°C show very little color change even with heating times up to 6 hours.

When the resin was heated without the woody component, no aromatic ν(C=C) bands were noted in the FTIR spectra even at temperatures up to 210°C. This finding is consistent with other reports that suggest that dehydrogenation leading to double-bond formation and formation of aromatic groups in resin acids is achieved at temperatures well over 200°C (Beck et al. 1998; Silvestre and Gandini 2008).

Table 2. Heating Experiments (Courtesy of Christina Bisulca and Brunella Santarelli)

Heating Temperature (°C)	Observations
90	Softened, no flow, sample hardens upon cooling due to loss of volatile component
100	Some flow, cools very rapidly, no significant chemical change based on FTIR
120	Golden yellow, flows easily but still viscous, no chemical change in FTIR
140	Starts to yellow slightly, flows readily
160	Flows readily, but is much more brittle when cooled, some chemical changes noted in FTIR
180	Golden brown/golden yellow, very fluid
200	Deep brown, very fluid

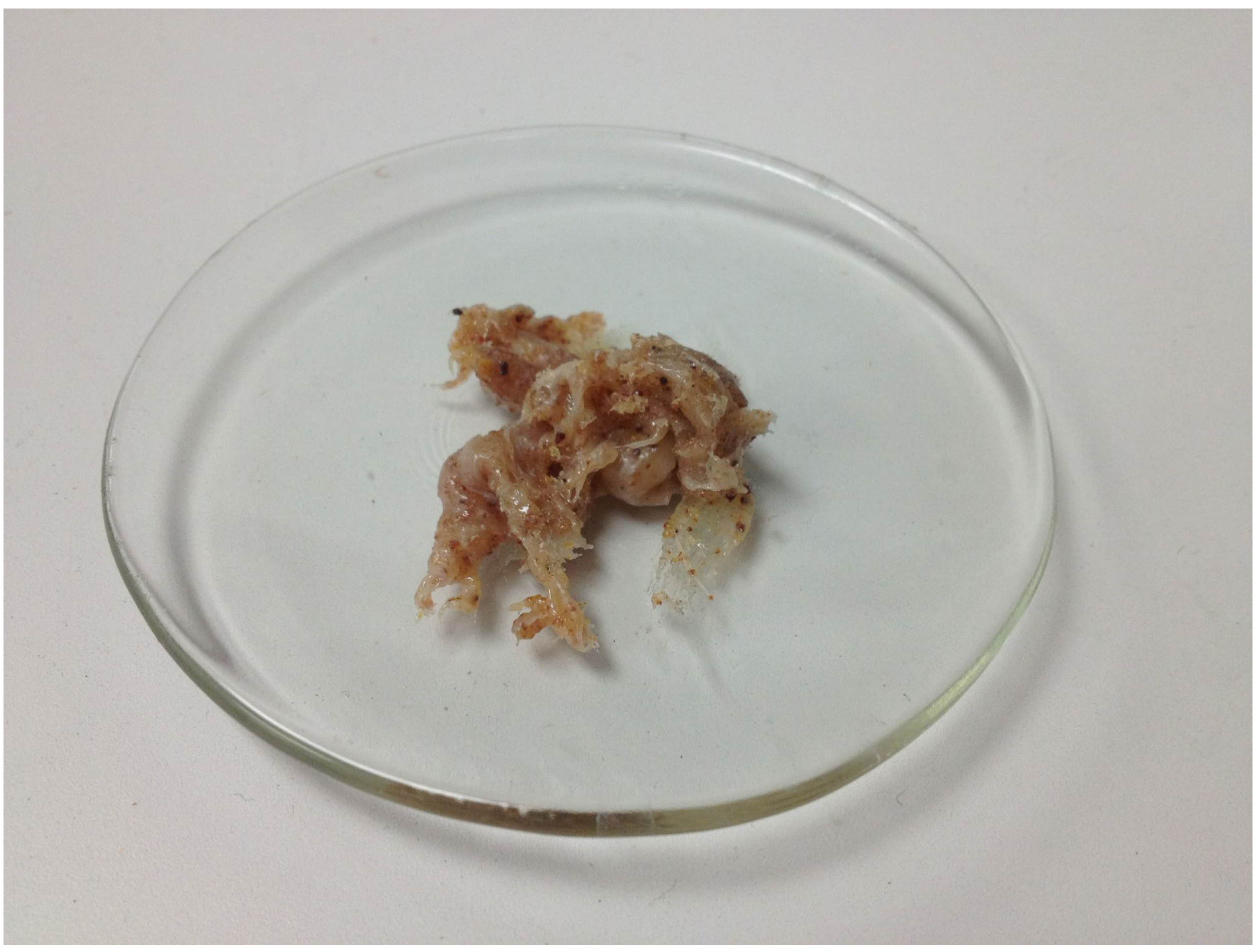

Fig. 16. Fresh resin as collected (Courtesy of Christina Bisulca)

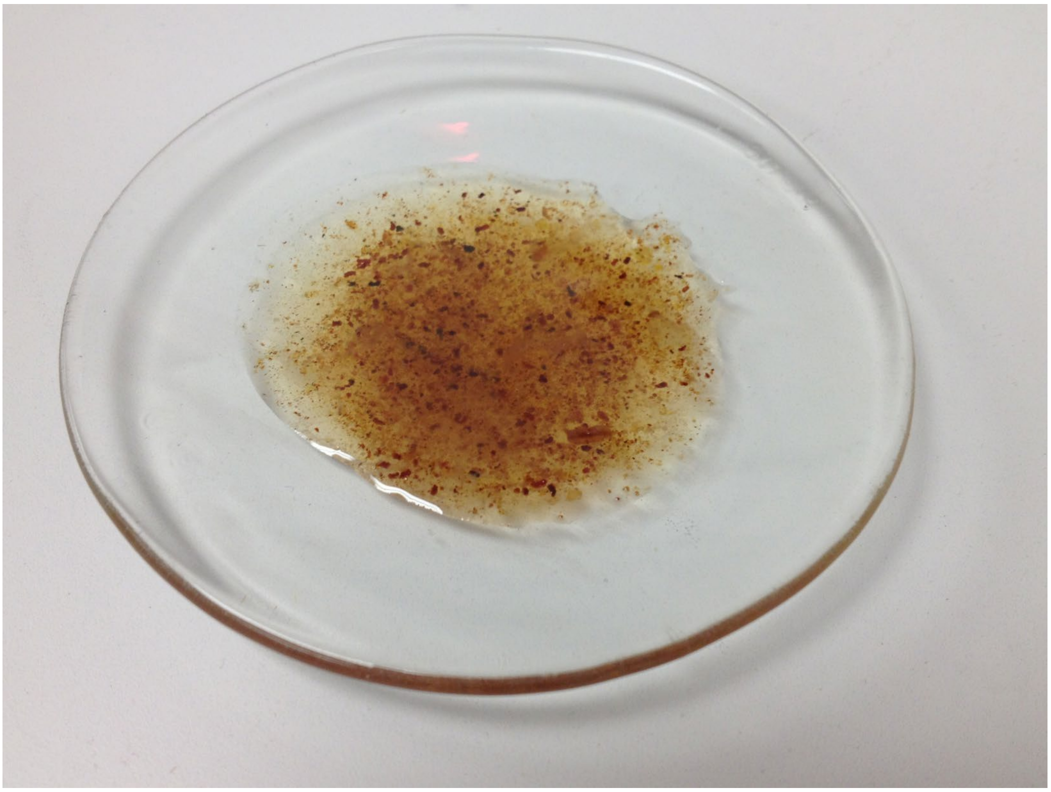

Fig. 17. Resin after heating to 90°C. Small particulates within the resin are pieces of bark or wood that were imbedded in the resin during exudation from the tree (Courtesy of Christina Bisulca)

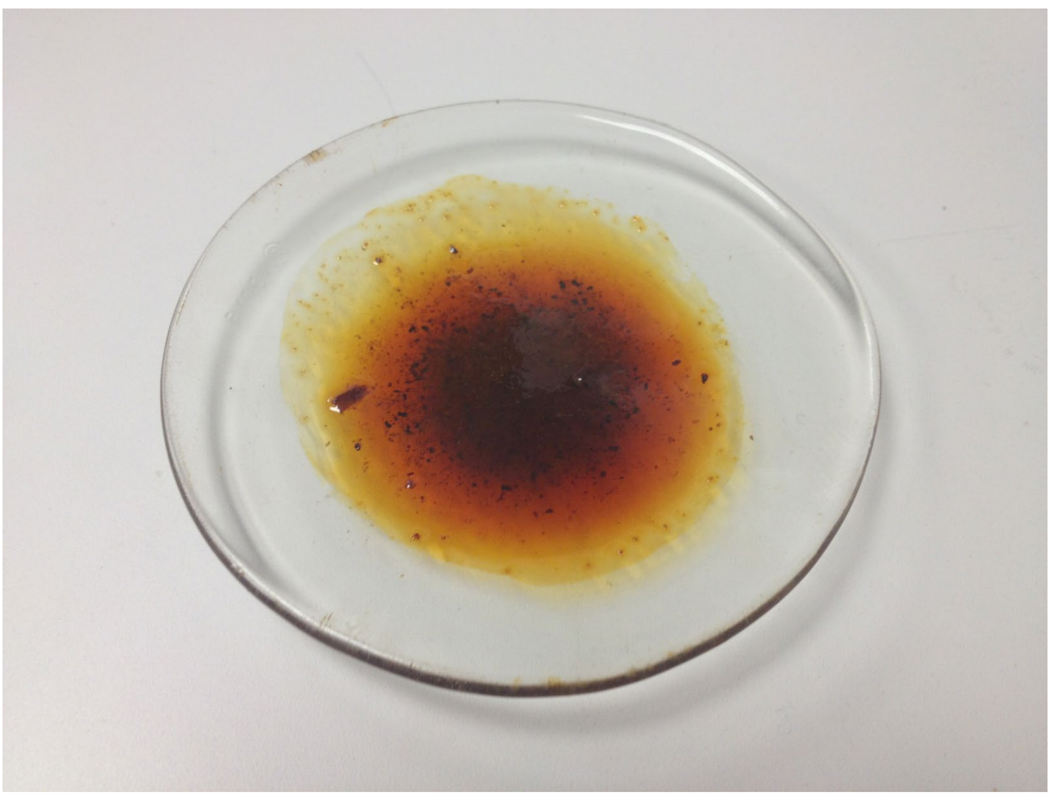

Fig. 18. Resin after heating to 200°C (Courtesy of Christina Bisulca)

FTIR analysis of the samples, however, showed increased oxidation upon heating, with the formation of a shoulder in the main carbonyl band at ~1725cm^{-1}, increased absorption of ν(C–O–C) bands in the 1200–1100cm^{-1} range, ν(−OH) in the ~3500–2500cm^{-1} range, and ν(C–O–H) at ~1240cm^{-1}.

When the resin was heated with the woody component present (as collected), significant changes were seen in the FTIR spectra. Most noteworthy were the formation of aromatic bands at 1515 and 1604cm^{-1} and the formation of a shoulder on the carbonyl band at ~1725cm^{-1}. Because these bands were not seen in the resin samples heated without a woody component, it is assumed that these changes are due to a reaction of the woody materials with the resin acids.

5.4 DISCUSSION

In the examination of the FTIR spectra of pitch basket samples, pitch coatings with a high aromatic component are typically dark brown to black opaque. Translucent yellow, red, to deep ruddy brown pitch basket coatings have a low aromatic component. On the basis of the heating experiments, it can be surmised that during manufacture, the darker pitch coating with a high aromatic component was heated at high temperatures with woody materials present in the resin. Conversely, pine pitch baskets with lower aromatic components were most likely heated with the wood material removed. In appearance, these coatings are translucent, and the color range from yellow to deep ruddy brown in these baskets is indicative of heating temperatures.

The formation of aromatic groups in processed pine resin is associated with *wood tar,* a product typically produced by heating highly resinous wood (Senftle and Larter 1988; Beck et al. 1998; Font et al. 2007). In our experiments, the reaction occurred even with minor contaminants from bark present in the resin as collected. The lack of an aromatic component in some pitch may suggest some form of processing of the resin before or during heating to preferentially remove the woody component, or else specialized collection techniques. Heat used in the technology of pitch baskets is described by several anthropologists (Tanner 1983; Simpson 2003), but a detailed description of the process is not given. In commercial production of pitch ("naval stores"), woody contaminants are removed by filtration (Panda 2008).

To confirm that the presence of wood during heating is the primary difference between these two pitch types, samples of each type were taken from pitch baskets for analysis. Figure 19 shows microscope images of pitch samples dissolved in ethanol. The sample taken from the dark opaque pitch with a high aromatic component shows that this pitch does indeed have a greater amount of woody contaminants (Fig. 19a, 19b).

Although the precise reactions produced by heating were not determined in this study, there were some unexpected results. The formation of aromatic bands in the resin itself normally associated with higher temperatures (over 300°C) was found to occur as low as 170°C when the resin was heated with woody materials present. The precise nature of the aromatic component noted in the FTIR spectra is currently under further investigation.

A concern noted during treatment was how the presence or absence of an aromatic component in pitch and the increased oxidation will affect its solubility. For example, when the darker, opaque pitch was observed to be less soluble and required more ethanol and increased pressure to readhere loose pieces, our experiments suggest that this is due to the difference in chemical composition of the pitch, which, in turn is related to the physical properties of the coatings.

6. CONCLUSIONS

Conservation surveys have evolved greatly over the years at the Arizona State Museum. They have moved from sequential steps of condition survey, proposal, analysis, experimentation, treatment to a more integrated approach. This project illustrates the value of combining survey, analysis, and treatment activities. The survey identified pitch baskets in an unstable condition prompting

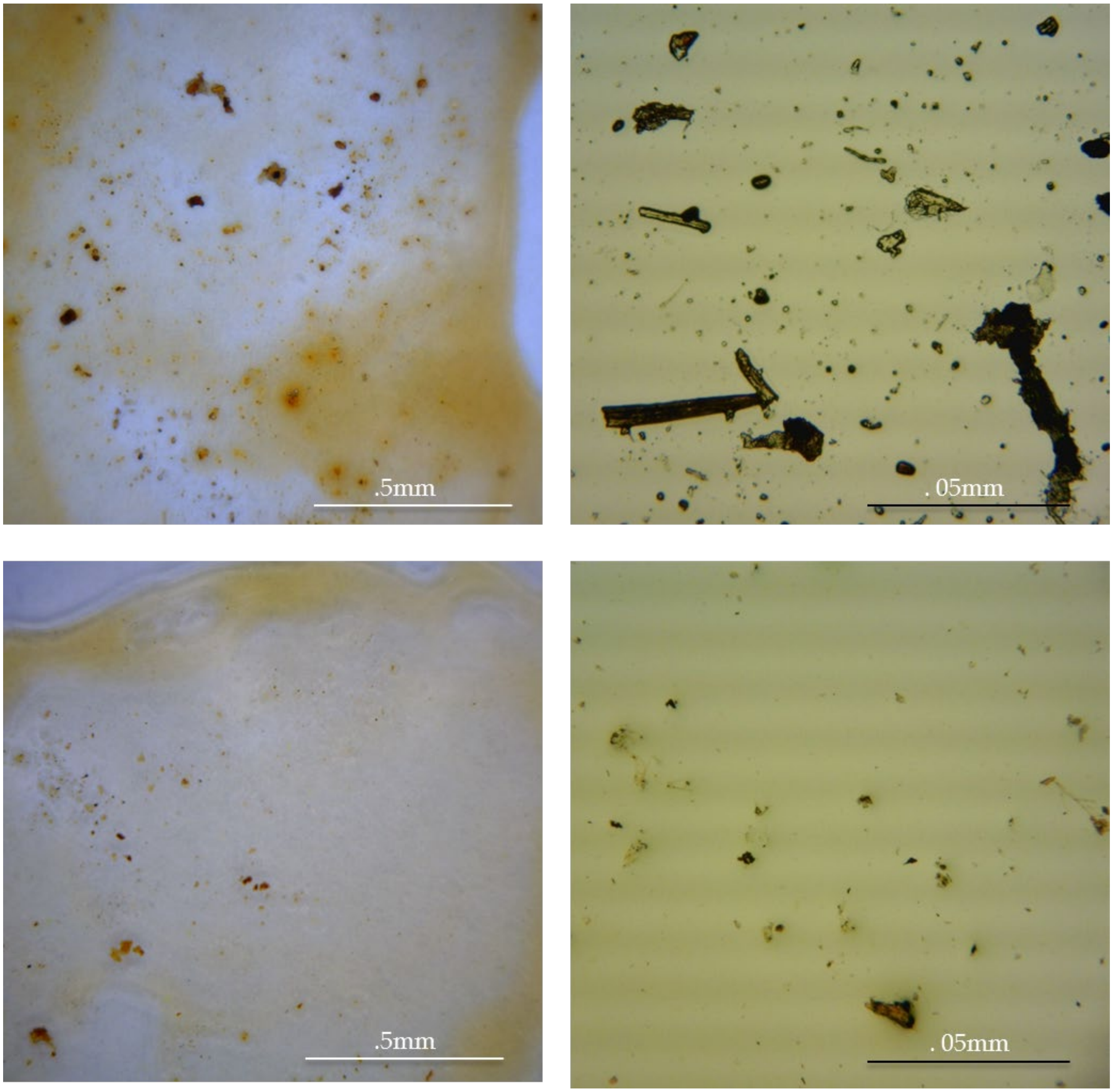

Fig. 19a. Sample of pitch dissolved in ethanol taken from a deep brown, opaque pitch coating with a high aromatic component in FTIR (ASM GP4344). 19b. High magnification image of (a) showing woody contaminants present in the pitch. 19c. Sample of pitch dissolved in ethanol taken from a light, translucent pitch coating with a low aromatic component in FTIR (ASM 1983-65-1). 19d. High magnification image of C showing minimal contaminants from woody materials. (Courtesy of Madeleine Neiman)

conservation treatment; the treatment required a protocol that would address ethical cultural and conservation concerns as well as questions related to chemical composition; the composition questions brought about analytical studies; the analytical studies supported the ethanol reactivation treatment protocol and provided information that clarified aspects of the lighter and darker appearance of pine pitch basketry technology. When allowed to be carried out in tandem, these activities serve to inform each other resulting in better care and understanding of collections. This project and its outcomes illustrate the benefit of conducting condition surveys–they highlight important preservation needs in a holistic way.

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