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RESIN AND LAC ADHESIVES IN SOUTHWEST ARCHAEOLOGY AND MICROCHEMICAL TESTS FOR THEIR IDENTIFICATION

CHRISTINA BISULCA, MARILEN POOL, AND NANCY ODEGAARD

The peoples of the Southwest used a variety of organic adhesives including pine resin and insect lac (shellac). A survey at the Arizona State Museum characterized over 100 artifacts with resinous materials or residues using Fourier transform infrared spectroscopy. Less expensive and more accessible methods—UV-induced visible fluorescence and microchemical testing—were also used for characterization and their accuracy was compared to Fourier transform infrared spectroscopy results. For pine resin, the Raspail test was used; for insect lac, a new microchemical test was developed based on the pH sensitivity of anthraquinone dyes present in insect lac exudates. Results show that microchemical tests are generally reliable even with archaeological materials. This is important as archaeological artifacts are aged and adhesives are often contaminated with burial accretions. By systematically evaluating these tests, further insights were gained. Most importantly, the Raspail test was found to indicate any terpenoid exudate and is not specific to pine resin. These results show that although microchemical tests continue to be useful, care should be taken when interpreting results.

KEYWORDS: Microchemical test, Raspail test, Southwest archaeology, Pine resin, Insect lac

1. INTRODUCTION

In 2011, the Arizona State Museum (ASM) was awarded a Save America's Treasures Grant for the conservation and rehousing of the Archaeological Perishable Collection. This collection has over 30,000 artifacts including basketry, sandals, wood artifacts, bows, arrows, textiles, cordage, vegetal artifacts, and botanical specimens representing 2,000 years of history in the Southwest. These artifacts sometimes contain plant and insect exudates as adhesives, like pine resin or "creosote lac," a shellac type excretion from lac insects endemic to the Southwest.

Despite the extensive use of these adhesive materials, there have been few studies for their scientific identification in Southwestern archaeological collections. The presence and identity of organic adhesives on artifacts in museums and repositories is typically not documented. In a search of ASM's museum collections database, only a single object was described as containing insect lac. Most adhesive materials were either not listed or were erroneously described as a plant "gum" or "pitch." Incorrect material identification and inconsistencies in nomenclature have been found in both the archaeological and ethnographic literature for the Southwest. "Gum" was the conventional or generic term used well into the 20th century to describe all resinous materials (Coville 1892; Kidder and Guernsey 1919; Pepper 1920; Castetter and Underhill 1935; McGregor 1941). However, the botanical definition of gum specifically refers to polysaccharide exudates produced by some plants in response to wounding (Langenheim 2003).

The conservation survey and storage upgrade presented an opportune time to examine the entire collection, allowing for trends to be noted across different cultures, artifact types, and time periods. The survey also assessed the use of non-instrumental methods—in particular, micro-chemical testing—for the analysis of adhesive materials. In conservation there has been a trend of using increasingly advanced analytical equipment and data processing for materials characterization in museum objects. However, there is a continued need for simple, reliable, and inexpensive methods that can be used without specialized instrumentation or while in the field. Such methods are particularly relevant with recently acquired archaeological collections undergoing processing through cultural resource management firms. This project evaluated the reliability of these less costly techniques by comparing results with those from FTIR.

2. BACKGROUND

Arizona archaeology in the period from ca. 100–1500 CE is dominated by three main cultural groups: the Hohokam, the Mogollon, and the Ancestral Pueblo (Anasazi). These groups occupied very distinct environmental regions. Consequently, the plant materials available to each group were distinct. Trends in adhesive use by each culture have already been noted in native pottery repairs (White et al. 2009).

The Hohokam were located in the arid Sonoran Desert in southwestern Arizona and northern Mexico. In this region, the main adhesive material available was insect lac (fig. 1). The lac insects that produce it are in the same family (Keriidae) as the lac insects of Asia that are used for the commercial production of shellac. In North America there are seven known species in the *Tachardiella* genus, which is only found in the Americas. The lac produced by these insects is often termed "creosote lac" because the species *T. larreae* hosts on the creosote bush (*Larrea tridentata*). However, the other species host on different plants (Kondo and Gullan 2011) and this term is somewhat of a misnomer. This excretion by the insects of the Southwest not yet been fully characterized, but other studies (Fox et al. 1995; Stacey et al. 1998; Derrick et al. 1999) and analysis at ASM indicates that it is similar to shellac, which is a polyester with a minor wax component.

The Mogollon in Arizona were primarily located in the dense pine and fir forests of the Mogollon Rim but also extended into the Chihuahua desert regions of New Mexico, Arizona, and north-central Mexico. The Ancestral Pueblo occupied the Colorado Plateau of the Four Corners region which is primarily shrubland and pinyon-juniper woodland (Plog 2008). For these two cultures, various pine resins are the most abundant adhesive material available (Bohrer 1973; Minnis 2010). The pine species used by these cultures is typically assumed to be pinyon pine because this tree is so highly resinous.

Pine resin is a water-insoluble terpenoid exudate secreted in response to injury (Langenheim 2003). Terpenoids are produced by conifers, and the types of terpenoids produced is varied depending on taxon. In most pine (*Pinus* spp.) resins, the predominant terpenoids are abietic and pimaric type acids (Langenheim 2003; Mills and White 2012). Although the resin used archaeologically is assumed to be pinyon pine, it is possible that it was obtained from other pine species or other conifers that are found in these regions, some of which produce terpenoid exudates (Mills and White 2012).

Anthropological and ethnobotanical sources cite other possible plant materials used historically. Mesquite gum is a commonly referenced material, particularly as a pottery repair adhesive, paint, and

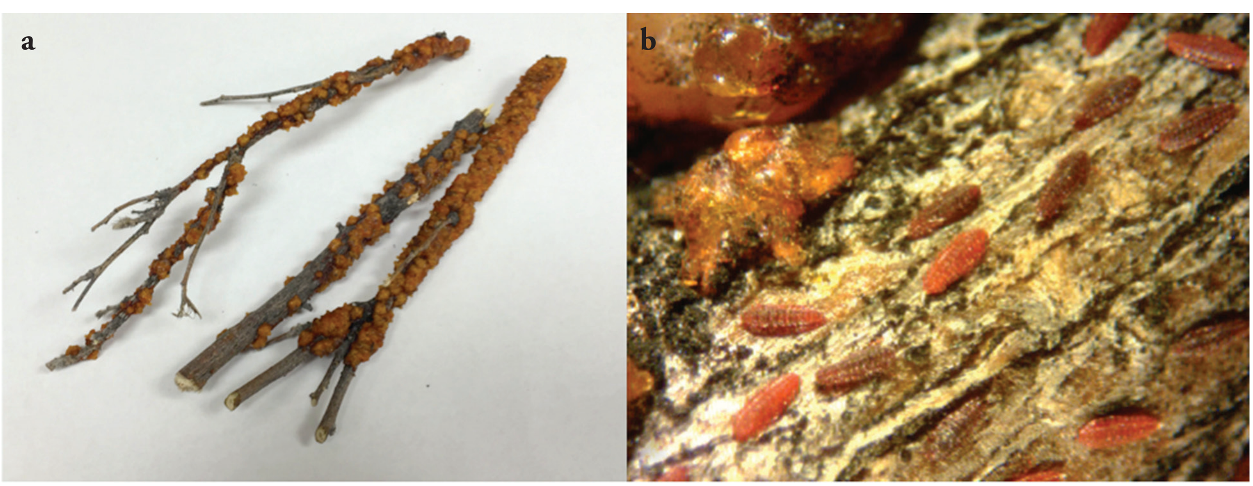

Fig. 1a. Insect lac exuded by the species *Tachardiella fulgens* hosting on the rosary babybonnet (*Coursetia glandulosa*) collected in Tucson, Arizona; 1b. Newly hatched lac insects on the specimen, left (*T. fulgens*). (Courtesy of Christina Bisulca and Marilen Pool)

adhesive in arrow manufacture (Castetter and Underhill 1935). There are also various starches and mucilages from cacti, seeds, and roots which could also have been used (Castetter and Underhill 1935; Odegaard 1997). Chemically, gums, starches, and mucilages are all water-soluble polysaccharides. However, polysaccharide materials have not yet been detected in a survey of archaeological repairs on pottery of the region in ASM's collections (White et al. 2009).

3. MATERIALS AND METHODS

Artifacts were initially examined under visible light and longwave ultraviolet radiation (365 nm). When organic adhesive materials were identified, they were sampled for analysis with Fourier transform infrared spectroscopy (FTIR) and microchemical tests. In total, 127 artifacts were sampled for analysis.

FTIR spectroscopy was performed on a Thermo Scientific Nicolet iS10 spectrometer with a Smart-iTR attachment, equipped with a HeNe laser and DGTS detector. Spectra were recorded in reflection mode, from 4,000 to 650 cm^{-1}, 64 scans at 4 cm^{-1} resolution. Identification was assisted by correlation with ASM's reference spectral database as well as other commercial libraries.

After identification with FTIR, select samples were also tested with microchemical methods to assess the effectiveness of these testing protocols. The "Test for Rosin using Sulfuric Acid (Raspail Test)" and the "Test for Complex Carbohydrates using o-Toluidine" were carried out as described in Odegaard et al. 2005. Due to the small number of samples found with polysaccharide material, the o-toluidine test is not discussed in this article. For updated protocols for using this test on anthropological collections and the interpretation of results see Bisulca et al. (2016).

For materials identified as pine resin, the Raspail test was performed. Using a spot plate, the sample was soaked in a saturated sugar solution for approximately 10 seconds. Excess sugar solution was removed and one drop of concentrated sulfuric acid (H_2SO_4) was added to the sample. A "raspberry red" color indicates the presence of pine resin. This color reaction with fresh pine resin will form in about one minute, but with older processed samples, the color can take up to 30 minutes to form.

To test for the presence of unprocessed insect lac, a new chemical test was devised. A sample about the size of the head of a pinhead was allowed to soak in two to three drops of 1.0M sodium hydroxide (NaOH). A purple color in the sample or surrounding solution indicates a positive for lac because the unprocessed exudate contains anthraquinone dyes that change color depending on pH.

This new microchemical test for insect lac is based on the fact that the insects of the Keriidae family produce dyes within their bodies and eggs that are orange to red. In *Kerria* spp. from Asia, this dye is extracted for commercial use and known as lac dye. The dye is composed of a group of related hydroxyanthraquinones known as the laccaic acids (Bechtold and Mussak 2009). Although the precise chemistry of the dyes from *Tachardiella* spp. has not yet been characterized, anthraquinones from shellac and other scale insects are chemically similar (Santos et al. 2015). The color of these anthraquinones is dependent on pH: for other insect anthraquinones (e.g. carminic acid, laccaic acids), the color will vary from orange to red in acidic solutions, to violet in alkaline conditions (Ketmaro et al. 2010; Bechtold and Mussak 2009). The surrounding lac exudate will swell and eventually hydrolyze in strong alkaline solutions (Limmatvapirat et al. 2005), which also helps to promote the dissolution of the anthraquinone dyes into solution for this test.

FTIR and microchemical testing was also completed on various reference materials for comparison. Insect lac from *T. fulgens* was collected on *Coursetia glandulosa* from Sabino Canyon, northeast of Tucson, Arizona, and *T. larrea* was purchased from the Colorado River Indian Tribes from the Lower Colorado River area close to Yuma, Arizona. Pinyon pine resin (*Pinus edulis*) was collected on the Navaho reservation approximately 30 miles west of Canyon de Chelly, Arizona. Additional terpenoid

Table 1. Raspail Test on Various Plant Terpenoids*

Exudate Type	Family	Species Tested	Color Reaction
Diterpenoid, Abietane/ pimarane	Pinaceae	*Pinus edulis, P. ponderosa, P. thunbergii, P. halepinus*	Red
Diterpenoid, Labdane	Araucariaceae	*Araucaria angustifolia*	Red
	Cupressaceae	*Cupressus arizonica, C. sempervirens*	Red
		Juniperus deppeana, J. scopulorum, J. virginiana, J. chinensis	Red
Triterpenoid	Burseraceae	Commercial Mexican copal (*Protium* or *Bursura* spp.)	Red, Brown
	Anacardiaceae	*Pistacia lentiscus, P. atlantica, P. chinensis*	Red, Brown
	Dipterocarpaceae	Commercial dammar	Red, Brown

*unless designated "commercial," all samples were collected directly from the plant source.

resins were collected from the University of Arizona Campus Arboretum (https://arboretum.arizona.edu/). Reference samples of pinyon pine resin and insect lac were heated in an oven and burned under flame to assess the effects of heat processing on microchemical tests and FTIR. For other reference gums and resins, samples were obtained from the Getty Conservation Institute Binding Media Reference Kit (Striegel and Hill 1997).

4. MICROCHEMICAL TESTS ON REFERENCE MATERIALS

The Raspail test is most commonly used for the qualitative determination of rosin (pine resin) size in paper by the procedure adapted by TAPPI (TAPPI 408). Although this test has been used for almost a century, the mechanism of the color reaction has never been published. Early references note that a red to purple color change is observed with other materials including proteins and fats. Due to the possibility of false positives, this study assessed other materials, in particular other plant exudates which may be encountered in artifacts. Terpenoid plant resins tested in this survey are listed in table 1.

Color reactions of different terpenoid resins using the Raspail test are shown in figure 2. In fresh samples, a color reaction was immediately observed on the surface of the sample. After several minutes, the red color reaction indicating a positive was visible in the solution surrounding the sample usually along with streaks of yellow and brown. This test was successful on all *Pinus* species tested. The test also produced positive results even after the resin was heated to 200°C (fig. 2, row B). This was tested because in anthropological samples it is assumed that the resin would have been heated prior to use, and heating pine resin to render it fluid has been documented ethnographically (Tanner 1982).

This test also resulted in a positive reaction for other diterpenoid resins. Pinaceae resins are primarily composed of tricyclic diterpenoids of the abietane or pimarane type (Dev 1989; Otto and Wilde 2001). Other conifers—such as cypress, sequoia, and juniper-like trees—typically produce dicyclic labdane-type diterpenoids, which are the most common terpenoid class found in resinous exudates

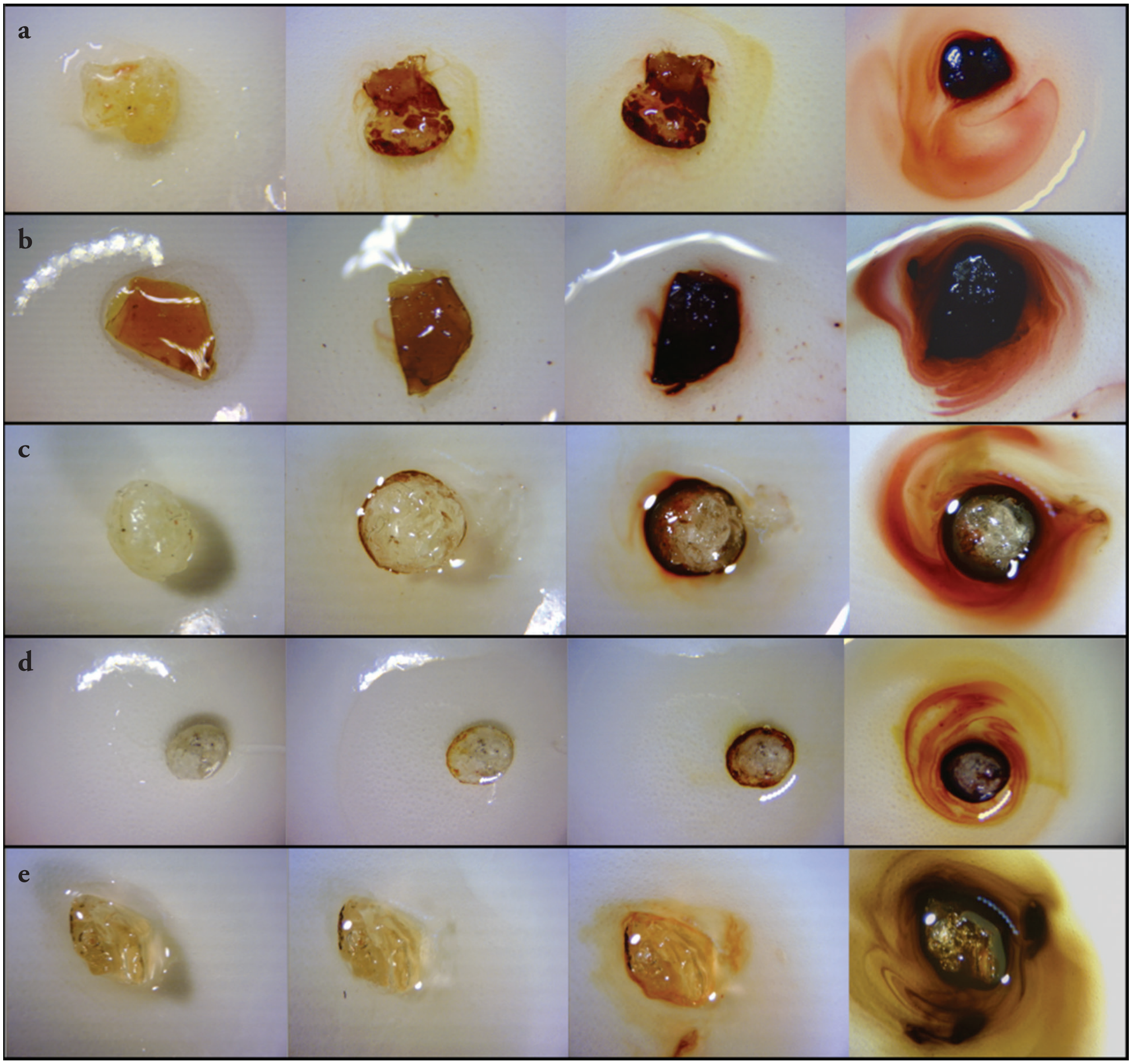

Fig. 2. Raspail test color reactions. Columns from left to right: before adding H_2SO_4 (conc.), immediate color reaction with H_2SO_4, color after 1 minute, color after 5 minutes. Rows: 2a. *Pinus edulis* (pinyon pine) exudate showing a typical color reaction; 2b. *Pinus edulis* after heating to 200°C; 2c. Labdane diterpenoid exudate from *Araucaria angustifolia* showing a positive color reaction; 2d. Triterpenoid resin from *Pistasia lentiscus* (mastic) showing a positive color reaction. 2e. Mesquite gum (*Prosopis glandulosa*) showing a negative reaction. (Courtesy of Christina Bisulca)

(Otto and Wilde 2001). In Arizona, this includes the Arizona cypress (*Cupressus arizonica*) and various junipers (*Juniperus deppeana, J. scopulorum, J. monosperma,* etc.) (Kearney and Peebles 1960). Like Pinaceae, these families are known to be highly resinous (Langenheim 2003) and their exudates could be encountered in artifacts. The labdane diterpenoids tested from Cupressaceae and Araucariaceae families all resulted in a positive red color reaction (fig. 2, row C). The labdane diterpenoids are the primary class of exudates that polymerize to form ambers (Lambert et al. 2008), and this explains why this microchemical test is also reported to work on ambers (Odegaard et al. 2005).

Triterpenoid resins (i.e., dammar, mastic) are also common plant exudates encountered in museum collections. Mexican copal is a triterpenoid resin that has been identified in archaeological artifacts

Table 2. NaOH Test for Insect Lac in Reference Materials

Sample	Appearance in UV	NaOH Test
T. fulgens, stick lac	Neon orange	Violet
T. fulgens, heated 200°C	Orange	Violet
T. fulgens, heated open flame	No fluorescence	Brown
T. larrea	Neon orange	Violet
T. larrea, heated 200°C	Neon orange	Violet
T. larrea, heated open flame	No fluorescence	Brown

(Stacey et al. 2006) and could potentially be encountered in ASM's collections. Triterpenoid resins were also found to result in a positive reaction with the Raspail test, although less consistently than the diterpenoid resins. Most samples showed an immediate red to orange color reaction at the surface, and sometimes produced a red to reddish-brown color reaction in the solution (fig. 2, row D).

As both di- and triterpenoids can produce a positive color reaction with the Raspail test, one should exercise caution in interpreting their results. A positive reaction can indicate any number of terpenoid exudates and is not necessarily diagnostic of abietic acid, a main component in most pine resins. Most controls tested (polysaccharides, proteins, waxes) produced a brown or yellow color in solution (fig. 2, row E). However, a red or violet color was also noted with certain proteins (albumin, egg yolk). In these cases, the color developed on the sample material only and not in the surrounding solution.

Microchemical test and UV results on reference samples of lac from Arizona are shown in table 2. The test using NaOH was successful on all fresh and heated lac samples. However, with excessive burning in open flame until charring occurred, samples no longer showed the color response. This would be expected, as the dyes are undoubtedly broken down in this process. It should also be noted that this test will not work on commercial shellac as the dye component is typically removed in processing.

5. ANALYSIS OF ARCHAEOLOGICAL MATERIALS

FTIR identification of insect lac and pine resin in archaeological samples is shown in figure 3. For tree resins, archaeological samples showed the best spectral correlation with Pinaceae resins based on FTIR. Further details on differentiation of the class of terpenoid exudates with FTIR are given elsewhere (Gianno et al. 1987; Tappert et al. 2011; Seyfullah et al. 2015).

In testing collections, the Raspail test gave a positive result for all materials determined to be pine resin with FTIR. Results were easiest to observe using a stereomicroscope, as archaeological samples often turned brown with areas of red positive reaction. When testing unknown materials, it is important to always compare results to known positive reactions (Odegaard et al. 2005). It should be stressed that this test will result in a positive red for other terpenoid-based tree exudates and does not necessarily indicate pine resin.

In testing archaeological collections, approximately 75% of insect lac resulted in a positive purple reaction to the NaOH test. In positive results, a purple color was visible in the testing solution or as purple areas within the resin when viewed under a stereomicroscope (fig. 4). Often the color change is

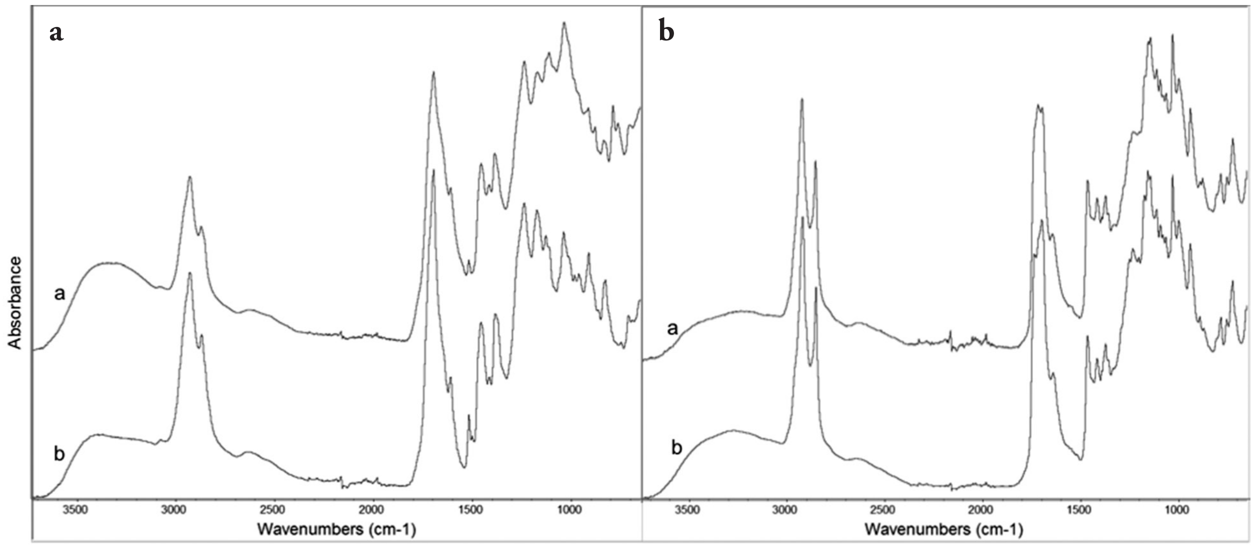

Fig. 3a. Pine resin sample from Ancestral Pueblo sealing ring, Cave X, cat no. A-14374 (a) and pinyon pine resin, ASM reference collection (b); 3b. Insect lac sample from Hohokam ball, Casa Blanca, cat. no. 2309 (a) and creosote lac (*Tachardiella larreae*), ASM reference collection (b). (Courtesy of Christina Bisulca)

visible only in localized areas of the sample as the dye is contained in the insects and eggs, not the lac exudate.

In cases where the NaOH test was not successful, the insect lac sample often appeared black, possibly due to charring. In these cases, because the sample itself was black this may have masked the color reaction. Lack of a color reaction may also be due to processing, as this material would have been heated during original processing. The insect anthraquinones that have commercial use (i.e. cochineal and lac dye) are known to be thermally stable (Fernández-López et al. 2013). However, burning the lac most likely results in the breakdown of these dyes. The anthraquinone dyes in insect lac are also somewhat water-soluble, so it is also possible that they have been lost, particularly at the surface where these objects were sampled.

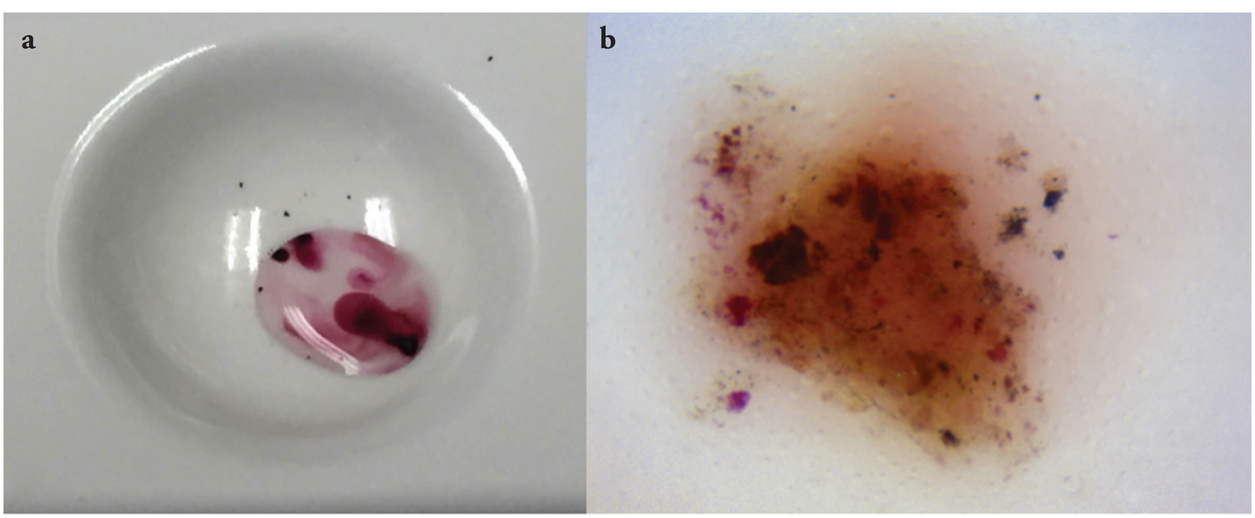

Fig. 4a. Spot test plate showing strong color reaction from an unprocessed lac sample from collections, cat. no. B-89, stick lac, Ventana cave; 4b. Microphotograph of sample showing purple color reaction in small areas. (Courtesy of Christina Bisulca)

Table 3. UV Identification in Collections*

Color	Insect Lac	Pine Resin	
Orange	10 (45%)	Green	13 (50%)
Green	3 (15%)	Orange	7 (27%)
No observed fluorescence	8 (40%)	No observed fluorescence	6 (23%)
Total	21 (100%)		26 (100%)

*Table lists the total number of artifacts tested and percentage of each by adhesive identified.

Because the collection was surveyed with UV radiation prior to analysis, it allowed for the assessment of reliability of identification using UV alone. While this type of examination is useful, it was not found to be reliable for the identification of these materials. This method gives positive identification only roughly half the time (table 3). More problematic is that this method can lead to incorrect identification, which in the assessment of collections at ASM could be as much as 25% of the time.

6. ADHESIVE USE IN SOUTHWEST ARCHAEOLOGY

The adhesive materials found in the archaeological artifacts of the Archaeological Perishable Collection defined by culture is shown in figure 5a. As expected, the adhesive materials found in Hohokam artifacts are predominantly insect lac and Anasazi predominantly pine resins. Surprisingly, the Mogollon used both adhesives, and approximately 60% of adhesives identified were insect lac. The use of insect lac in the Mogollon and Ancestral Pueblo regions has not been previously identified. For the complete results of this adhesives survey see Bisulca et al. (2017).

Several artifacts were found to have a polysaccharide based material. It is not possible to determine the precise identity of these materials (plant gum, starch, mucilage, etc.) based on FTIR alone. This is a particular problem with archaeological materials as they often are contaminated with soils, clays, or other aluminosilicates that overlap with bands characteristic of polysaccharides. However, these artifacts may indicate the potential use of plant polysaccharides like mesquite gum or seed and root starches (Odegaard 1997).

Some artifact groups demonstrate possible selection of these adhesives based on their material properties. The use of insect lac and pine resin in the Mogollon arrow foreshafts is shown in figure 5b. Both pine resin and insect lac were found used in this artifact group. However, as a hafting material, primarily insect lac was found. On three arrow fragments (A-17168-x3, x8, x9), both materials were used in the same artifact where insect lac was used in the hafting and pine resin in the tenon joint (fig. 5c). Insect lac appears to be the preferred material for hafting among the Mogollon. The preference for insect lac is likely because it is a stronger, less brittle material than pine resin, making it a superior material for this particular application. This example shows the cultural significance of adhesives and why identification of these materials is important to understanding collections.

7. CONCLUSION

This survey of adhesive materials in the ASM's perishable collections showed that both insect lac and pine resin were used by each of the main cultural groups represented in the archaeological record of Arizona.

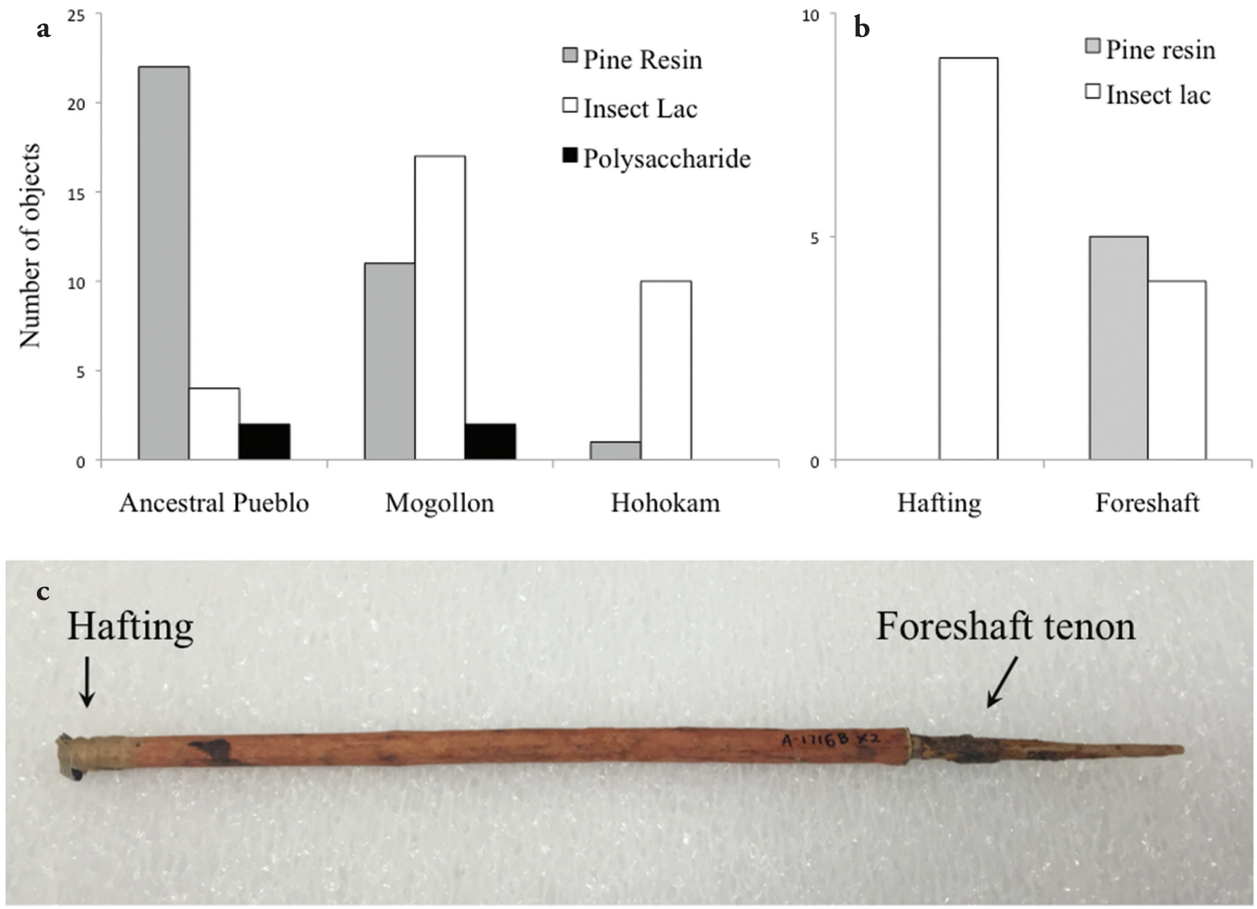

Fig. 5a. Objects with pine resin, insect lac or polysaccharide adhesives in the ASM Archaeological Perishable Collection by culture; 5b. Use of adhesive materials in Mogollon arrow foreshafts; 5c. Arrow foreshaft, Mogollon, Red Bowl Cliff Dwelling, 1325-1400 CE, A-17168. In the arrow pictured insect lac was found in the hafting and pine resin on the tenon joint. (Courtesy of Christina Bisulca and Marilen Pool)

Results highlight that historic terminology and catalog information can be misleading for the identification of these types of organic materials. Through this research, conservators updated and improved the collections records and also increased understanding of the collections, not only for preservation but for deeper comprehension of the material culture of the Southwest. Non-instrumental methods—in particular, microchemical testing—was found to be reliable showing these methods continue to be great benefit for understanding collections. Although UV analysis can be helpful in the identification of adhesives, results are not accurate and, more importantly, are often misleading.

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