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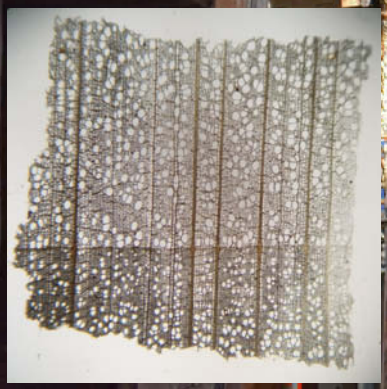

Wooden Artifacts Group Postprints

Presentations from the 2019 AIC Annual Meeting in New England
Wooden Artifacts Group Sessions

Wooden Artifacts Group

Postprints of the

Wooden Artifacts Session

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AMERICAN INSTITUTE FOR CONSERVATION OF HISTORIC AND ARTISTIC WORKS
47TH ANNUAL MEETING, NEW ENGLAND, 2019

POSTPRINTS OF THE
Wooden Artifacts Session

INDEX

YURI YANCHYSHYN

Furthering Wooden Artifact and Architecture Conservation in Ukraine on a Fulbright

ABSTRACT—The Fulbright Scholar Program offers conservators the opportunity of sharing their knowledge and experience in distant lands. In spring 2018, under the auspices of the Fulbright Specialist Program, I spent 6 weeks introducing wooden artifact conservation to the students of the Department of Architecture and Conservation at the Lviv Polytechnic National University, Ukraine. This presentation illuminates my experiences, the little-known aspects of Ukraine's heritage of wooden architecture and artifacts, and the advocacy for conservation and conservation education that became an integral aspect of my visit.

After running a successful furniture conservation private practice for several decades in New York City, I realized I wanted to broaden my horizons. This desire led to my applying for Fulbright Specialist Status in 2016. What I did not know at that time was that my yearning for a small diversion would change my life in unpredictable ways.

The Fulbright program centers on countries, and I initially focused on Ukraine because I am of Ukrainian ancestry and fluent in the language, even though I have never visited the land of my parents. After many inquiries and connections, I found myself having frequent Skype conversations with Professor Mykola Bevz, PhD, head of the Department of Architecture and Conservation at the Lviv Polytechnic National University in Lviv, western Ukraine. Professor Bevz was looking to expand the department's existing stone conservation program to include the conservation of wooden architecture and artifacts as well. He desired to educate his students utilizing a Western model so that they would be able to preserve Ukraine's UNESCO World Heritage Site wooden *tserkvas* (or "churches" in Ukrainian), as well as other wooden architectural monuments. He had been the guiding force in spearheading their UNESCO designation and felt that the expertise for preserving them was not available in Ukraine. After several such conversations, he extended an invitation and successfully applied for Fulbright funding. My journey had begun.

The West knows little of Ukraine's rich artistic heritage, such as its tserkvas—vernacular ecclesiastic architecture built of wood. There are approximately 3000 surviving examples, from the 16th through the 19th centuries. They are distinguished by their highly skilled joinery; innovative structural solutions used in their construction; a wall of icons that separate the sanctuary from the nave, called an *iconostasis*; and their stylistic identity and painted interiors. Over the centuries, these tserkvas were cared for by local craftsmen, who were well versed not only in their craft building traditions but also in their maintenance. However, the 20th century was a major disruptive force, with its wars, border changes, population migrations, and purposeful disuse. Today, although the majority of these tserkvas continue to function for their original purpose, two general factors account for their current condition: persistent neglect during the time of the Soviets and improper restorations.

During a winter visit, as well as during my 6-week spring Fulbright stay, I visited several of these churches. One example is the UNESCO-listed Tserkva of the Descent of the Holy Spirit in Potelych, built in 1502 (fig. 1). It is the oldest surviving tserkva in the Lviv region. This tripartite ground plan church is unstable on its foundation due to its site orientation, built into a sloping hill. Over centuries, this has affected the stability of its laid stone foundations, necessitating temporary wooden structural posts and beams to keep it from collapsing. In addition to the early date of its construction, this tserkva's unique value is its painted iconostasis, dated to 1628. Typically, one would find a multirow carved and constructed wall of icons that separates the nave from the sanctuary. In this early case, however, the upper tiers of the iconostasis depict a painted version of such a wall, executed on the tserkva's interior structural members. What makes it especially interesting is that there is an earlier version of a painted iconostasis beneath the visible one, seen in areas of paint loss. This painted iconostasis would be an excellent candidate for infrared spectroscopy analysis to determine the extent of the images hidden beneath the visible version. In addition, paint losses and paint delamination are prevalent on the interior horizontal timbers (fig. 2). The same situation also exists on the carved iconostasis.

Another example is the 16th-century UNESCO-listed Tserkva of St. George in the city of Drohobych (fig. 3). On the day of my visit, the temperature was 31°F, the relative humidity was 46%, and there was snow on the ground. These

Fig. 1. Tserkva of the Descent of the Holy Spirit, Potelych, Ukraine, ca. 1502.

environmental readings emphasized to me that these tserkvas have survived for centuries in a completely unregulated environment. It is in much better condition than the tserkva in Potelych, due to the care it received over the decades and is now a museum. The interior painting is extensive, even up to the cupolas. Even so, a cursory overview also revealed large areas of paint loss and delamination, once again especially prominent on the interior faces of the horizontal timber walls. This paint application method presents challenging stability issues. The horizontal structural timbers absorb moisture on their outer faces, which permeates through to the interior painted surfaces, thereby contributing to the delamination of the paint layers. Other areas of concern were the splits in the icons and the large iconostasis, as well as previous restorations, such as the bronze powder overcoats on the iconostasis columns that have now oxidized. Because water infiltration is a significant concern regarding the stability of these tserkvas, it was heartening to note that the wood shakes on the roof had recently been partially replaced, utilizing softwood timbers, riven in the traditional manner.

The UNESCO-listed Tserkva of the Holy Trinity in Zhovkva, built in 1720, also has structural issues, just like the one in Potelych. The southeastern side of the tserkva is sagging, as evidenced by the distorted frame of the panel painting. This tserkva was raised and leveled in summer 2019. A visual examination of the iconostasis revealed an original silvered layer, with extensive craquelure, covered with an orange glaze overcoat, typical of the 16th through the 18th centuries in that region. However, many other sections revealed a very smooth gold-like covered surface, almost like gold paint. I could not verify these initial observations, as a copy of the treatment report was not available. These surfaces would be a prime candidate for testing, which would reveal the nature of the overcoating, and whether it covers any original silvered and glazed surfaces. A close inspection of the horizontal timbers of this tserkva, as in the case with many

Fig. 2. Paint losses, interior timber walls, Tserkva of the Descent of the Holy Spirit, Potelych, Ukraine, ca. 1502.

tserkvas, revealed extensive insect infestation, a condition that also afflicts many of Ukraine's wooden artifacts.

This condition became evident when I visited an art object repository in a former Capuchin monastery. I discovered an unregulated environment regarding temperature and humidity holding more than 1200 renaissance and baroque polychrome sculptures, many hundreds of icons and paintings on canvas, and an undetermined amount of works of art made of metal, including furniture items. They had been collected by Borys Voznytsky, a courageous curator, who was active during the Soviet period in the 1960s and 1970s, when many of these tserkvas, Roman Catholic churches, and synagogues were closed and turned into

Fig. 3. Painted and gilded iconostasis—Tserkva of St. George, Drohobych, Ukraine, 16th century.

storage facilities, and their objects discarded. A cursory visual inspection revealed that all of these objects are in various states of instability, some with significant losses and infested with wood-boring insects, like the timbers of the tserkvas. The repository registrar informed me that large numbers of these insects populate the building during summer months. The current approach is to inject a toxic insecticide into each flight hole, cover the art object in plastic, and wait for some time. This procedure can pose a serious human health hazard. One significant result of Voznytsky's collecting efforts was the discovery of the work of the baroque sculptor Johann Georg Pinzel. The Louvre Museum subsequently mounted an exhibition of his work in autumn 2012.

A visit to the Lviv Polytechnic National University, where I was to teach, introduced me to a different situation. Founded in the mid-19th century by the Austrian-Hungarian monarchy, it has a magnificent interior, and its Assembly Hall was in the process of restoration. Students were manually stripping numerous paint overcoat layers and in the process damaging the surfaces. Hoping to suggest an alternative approach, I inquired about the hall's coating analysis report. I learned that in this case the document did not exist, and the analytical results, relying on straightforward microscopy with no differentiation of coatings layers, were communicated verbally. When I then inquired about performing my own preliminary solvent solubility testing, I realized that would not be possible, as the department did not have a conservation laboratory that could provide me with the necessary solvents.

Also located within the building was the faculty library, with beautifully executed woodwork and ceiling. Unfortunately, the Soviets had removed the original coating, and the surfaces appeared to be bare wood. I was asked to provide recommendations regarding restoring the appearance of the original coating. Luckily, I discovered a section of the library that retained its degraded, although likely original, surfaces, so samples could be taken and analyzed. However, I realized that I was up against the same stumbling block as with the Assembly Hall—no lab, no instrumentation, and no personnel to run it equals no analysis or recommendations.

Interspersed with all of this were brief visits to student classrooms and workrooms, where I did not see any microscopes. It was at that point I realized that if the ambition of Professor Bevz

and his faculty were to preserve Ukraine's cultural artifacts utilizing a Western model, much more was needed than my coming for 6 weeks, giving a couple of lectures and teaching a few workshops.[1] What was truly needed was the adoption of a more scientific attitude toward conservation, an upgrading of their conservation curriculum, an implementation of a code of ethics, and ultimately the realization that art conservation is a profession.

To further that end, even before my formal Fulbright journey was to begin, I suggested that a visit to the conservation institutions in the United States would be beneficial so that our guests could see for themselves examples of conservation education and practice. This thought led to Myron Stachiw, architectural historian and the former director of the Fulbright program in Ukraine, and me to co-organize such a tour for Professor Bevz and his fellow faculty member, Maryana Kaplinska, PhD, in autumn 2017. Our guests visited and gave presentations informing Western audiences of Ukraine's cultural heritage at the following institutions: New York University's Institute of Fine Arts Conservation Center, University of Pennsylvania's Department of Historic Preservation, University of Delaware's Winterthur Museum Conservation Lab, and University of Amherst's graduate program in Historic Preservation. They also toured the Metropolitan Museum's conservation labs; Museum of Modern Art's conservation labs; Howard L. Zimmerman Architects; University of Pennsylvania's Museum of Archeology and Anthropology conservation labs; and Columbia University's Graduate School of Architecture, Planning, and Preservation. They further visited the University of Delaware's Center for Historic Architecture and Design; Smithsonian's Museum Conservation Institute; Museum of Fine Arts, Boston's conservation labs; Peabody Essex Museum's conservation labs; and the National Park Services Northeast Region Historic Architecture, Conservation, and Engineering Center. They also called on Historic Deerfield, Handhouse Studio, Jablonski Building Conservation Inc., George Washington's Mount Vernon, and Harvard's Ukrainian Research Institute. These visits were beneficial because our visitors learned of various conservation approaches and the diversity of technical expertise in the United States.

In addition, realizing that it would be helpful for our colleagues to have analytical reports that could serve as models for their future analytical work, I reached out to Richard Wolbers of the University of Delaware, Winterthur Museum Conservation Program, who generously provided stratigraphic coatings analysis of the Lviv Polytechnic Assembly Hall samples. Adriana Rizzo and Frederico Caro of the Metropolitan Museum's Department of Scientific Research provided coating sample analysis of the Lviv Polytechnic Faculty Library so that a replacement coating could be formulated. For the teaching component of my Fulbright Specialist visit, I developed a comprehensive series of nine lectures forming an introduction to the conservation of wooden artifacts. The topics included were the following: What Is Conservation? Its Principles, Procedures, History, and Ethics; Examining Works of Art and Condition Reporting; Wood—Its Structure and Properties; Wood Identification; Joinery–Construction–Panels; Adhesives and Grounds; Transparent Coatings; Colored and Painted Wood; and Aging and Degradation. When my Fulbright began in spring 2018, I felt fully prepared to make a difference.

My lectures were written out in English, then translated and delivered in Ukrainian, as most of my conservation students' English language skills were modest. Strengths that the students did have, however, whether at the Lviv Polytechnic National University or the National Academy of Fine Arts in Kyiv, were superb eye and hand skills. These skills became evident during a student project on the conservation of a 19th-century wood chandelier. The students quickly progressed from examining an art object and preparing a condition report to consolidation techniques and gesso in-fills. They became genuinely excited when I introduced them to microscopic wood identification, utilizing a microscope that I had brought with me. During our classroom interaction, as well as during museum visits, I introduced English language skills and conservation terminology.

Realizing that the science portion of their education required strengthening, I lobbied for the introduction of 2 years of chemistry in their bachelor's degree program and a separate year of advanced materials analysis in their master's degree program. And when I learned that their department was creating a conservation lab, I donated the digital microscope I had brought with me.

Another aspect of my visit also came into focus, which was the broader attitude toward historic preservation. I became concerned when I realized that although Ukraine has strong landmark preservation laws, the government was not enforcing them due to the deep corruption of the judicial system, which in turn then led to pressure on the faculty. In light of this and seeing the general neglect of architecture and works of art, I decided to step outside of my prescribed role as a visiting lecturer and to speak up at conferences and meetings supporting historic preservation. One of many examples was my presentation at a meeting of the preservation architects, archaeologists, and restorers of the City of Kyiv to address the fate of controversial archaeological excavations of Kyiv's earliest 10th- to 11th- century settlement area close to the banks of the Dnipro River. Archeologists had uncovered the remains of an 11th-century log roadway and numerous building sites. However, a proposal had been put forth to create an underground shopping mall on the site instead. I lent my voice in support of preserving the remains, having the archeologists continue with their research, and building a museum to exhibit their discoveries. Of particular concern were technical questions regarding the preservation of the archeological wood. I also gave several interviews with the press, culminating in an address to Ukraine's Parliament (fig. 4) advocating for increased funding for historic preservation.

Fig. 4. Presentation at Ukraine's Parliament.

Ukraine is an old nation, although relatively new as an independent and sovereign post-Soviet state. It chose the West during its Revolution of Dignity in 2014 and is in the process of redefining its identity, reexamining its history, and reassessing its cultural heritage and how best to preserve it. The Fulbright program permitted me to become aware of this process; to meet the faculty of the Lviv Polytechnic National University, and others, who are on the front lines of this process; and in a small way to become a part of it. It was a life-changing experience.

END NOTE

1. The Ministry of Culture, a holdover from the time of the Soviets, manages all aspects of public museums and historical and architectural landmarks. It also supervises the conservation of works of art and the training and certification of conservators and restoration specialists. Specialized certification is necessary for a conservator to be able to treat various works of art throughout the country. This highly centralized and outdated approach has created several shortcomings: a knowledge gap due to the lack of fluency in English, a reluctance to incorporate a greater degree of science in the conservation curriculum that then impacts potential research, an insular approach to the choice of an institution's staff, and a resistance to treatment innovations common in the West. This has led to young conservators and conservation students agitating for change.

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Forestry University; Ihor Kozhan, director of the Andrey Sheptytsky Lviv National Museum; Yuri Ostrovsky and Larissa Razinkova Voznytska of the Lviv National Academy of Fine Arts; and Olha Kotscovska, director of the Ivan Trush Lviv College of Decorative and Applied Art. Special thanks to Dmytro Ostapiv, head of the Laboratory of Molecular Biology and Clinical Biochemistry, the Institute of Animal Biology NAAS, for lending his laboratory's Zeiss Axioscope so that it could be utilized in my lecture on microscopic wood identification. Last, a heartfelt thanks to Myron Stachiw, who assisted me in ways too numerous to mention and made this Fulbright experience possible.

AUTHOR BIOGRAPHY

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KAROLINA SOPPA, KEVIN KOHLER, ELISA CARL, ELECTRA D'EMILIO AND THOMAS GEIGER

Strengthen Methyl Cellulose with Nanocellulose for High Relative Humidity

ABSTRACT—In this article, we discuss the strengthening of methyl cellulose with nanocrystalline cellulose and microfibrillated cellulose for high relative humidity (75%, 84%, 97%) during re-adhesion of chalk ground. Methyl cellulose is the least hydrophilic cellulose ether among the water-soluble ones. Its long-term stability is very high. It has good tensile strength for re-adhesion of flaking paint on canvas or wood. Nanocellulose has been recently proposed as a novel consolidant for canvas and paper consolidation, as well as a reinforcement for some consolidants. Our final mixtures were more resistant to high humidity (75%RH), than pure methyl cellulose and showed that ratio, homogenizing, and application methods were crucial.

1. INTRODUCTION

Methyl cellulose (MC) meets many of the requirements of conservators. It has both polar and nonpolar groups, and it is the most hydrophobic of the water-soluble cellulose ethers. It is compatible with both polar and nonpolar media in an object, and the resistance to higher humidity should be better than other water-soluble cellulose ethers.

MC has high aging stability concerning relative humidity (RH) fluctuations and light, reworkability, cohesion, and adhesion lower than the substrate. Last but not least, it is recognizable as a new ingredient (production of MC approximately since 1950). Its long-term stability is very high, according to Feller and Wilt (1990). A good tensile strength for re-adhesion of flaking paint on canvas or wood (fig. 1), as well as for wood gluing, has been proven for MC by some conservators (Döll 1997; Mercier 2003; Sindlinger-Maushardt and Petersen 2007; Soppa et al. 2014). Therefore, the use of MC is becoming more common in the field of conservation. However, the cohesion decreases dramatically for free films when the relative humidity is rising (Debeaufort and Voilley 1997). In the range of 22% to 53%RH, the difference in tensile strength was only 9%, whereas for 75%RH, the decrease amounted to 46% and for 84%RH even 80%. Considering that adhesion depends on substrate, one would have to ask what happens in different adhesive joints and if it leads to a weak adhesion, and, furthermore, how to make MC less sensitive to high relative humidity. After all, most of our heritage is located in churches, castles, collections, or museums without climate control systems, and many have a constant high humidity.

Cellulose is a linear homopolysaccharide of β-1.4-linked anhydro-d-glucose units. The glucose units with three hydroxyl groups interact strongly intra- and intermolecularly via hydrogen bonds forming crystalline units that are linked together by amorphous domains. The modulus of elasticity of the perfect crystal of native cellulose was estimated between 130,000 and 250,000 kN/cm^2. The tensile strength was assessed to be in the range of 800 to 10,000 kN/cm^2.

Therefore, the question of whether the strength of cellulose nanofibrils (CNF) or cellulose nanocrystals (CNC) can be used for bonding is justified. Furthermore, CNC and CNF have recently been proposed as a novel consolidant for canvas and paper consolidation, as well as a reinforcement for some consolidants at room temperature and up to 60%RH (Cataldi et al. 2015; Bridarolli et al. 2018; Nechyporchuk et al. 2018).

After drying, unmodified CNF and CNC are not redispersible in water because of hornification. If manufactured from sulfuric acid, dried CNC is water redispersible because of surface functionalization with sulfate groups. Irreversible hornification is not in accordance with our professional requirements regarding retreatability or reversibility. However, the adhesive mixtures of MC with the nanocelluloses are further water soluble after drying because of the water-soluble MC. Consequently, mixing the components MC and CNF or CNC could lead to a water-soluble yet more resistant adhesive at high relative humidity. CNC has the advantage of strong tensile strength, and considering that it has only crystals, it should be less reactive to relative humidity fluctuations. For fluctuations between 22% and 60%RH, CNC gave the lowest mechanical changes upon relative humidity variations (Bridarolli et al. 2018; Nechyporchuk et al. 2018). Hence, we tested the ratio of the components (for preparation, see Kohler et al. [2018]), the viscosity, the film properties, and, finally, the behavior of the mixtures in a chalk ground glue joint at high humidities (75%RH, 84%RH, and 97%RH). Several analytical techniques were used for the product characterization (tensile strength tests, elasticity tests, weighing technology). Subsequently, we analyzed the penetration into porous chalk ground by means of fluorescent MC and thin sections.

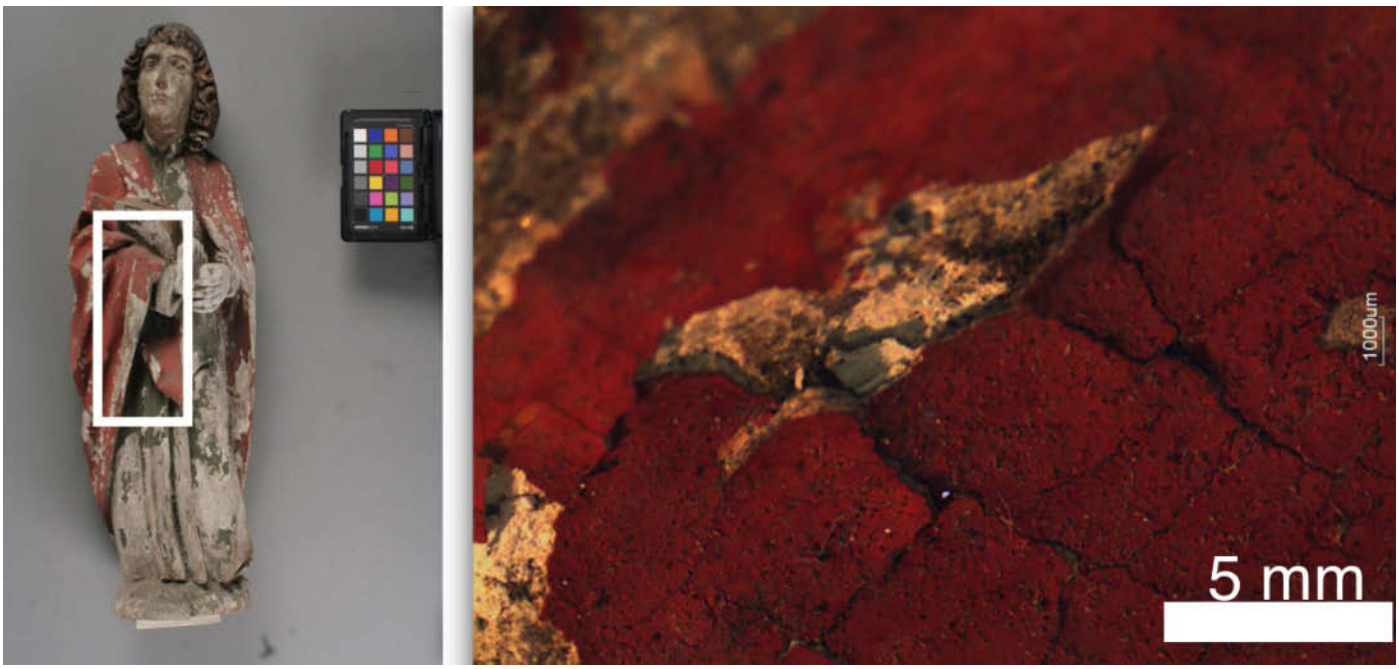

Fig. 1. Sculpture of St. John (16th century) and a detail of the cohesive failure in the chalk ground. Courtesy of Manon Léchenne.

Here we present only the results of the viscosity and tensile strength tests.

2. MATERIALS AND METHODS

2.1 ADHESIVES

As MC, Benecel A4C with DS of 1.8 from Aqualon was chosen. The fibrils forming the CNF network had individual diameters of about 3 to 100 nm and a length greater than 1000 nm. They were produced at Empa by disintegration of cellulose pulp in an ultrafine friction grinder. The CNF is produced from suspension of 2 wt% of bleached cellulose pulp and subsequently dewatered to 8 to 10 wt%. To obtain CNC, CNF can be further processed with sulfuric acid (H_2SO_4) or hydrochloric acid (HCl). The acid hydrolysis removes the noncrystalline areas, and only the crystalline areas remain. For an overview of the adhesives and their properties, see figure 2.

A suspension of CNC 7 wt% in water forms a mechanical stable gel. The CNC is mixed with water, heated to 60°C, and stirred for 15 minutes. Continue stirring as long as it is turbid. CNC is commercially available from The University of Maine with 0.85 wt% sulfate groups on the crystal surface. These ensure a stable suspension without the need for addition of surfactant.

For the mixtures of MC with CNF and CNC, respectively, both former pretests (see Kohler et al. [2018]) and new tests revealed that the fastest and most reliable mixing method can be performed by a SpeedMixer at 3500 rpm for 30 seconds.

The adhesive strength pretests for resistance to 75%RH with two mixtures (MC 3 wt% and CNC 1.5 wt% or CNF 1.5 wt%, 1:1) indicated that CNC did reduce the sensitivity of MC, whereas CNF showed little effect. This correlates with the results of Bridarolli et al. (2018) and Nechyporchuk et al. (2018), who tested by 60%RH. Therefore, only mixtures with CNC were investigated further. The concentration of the individual components and the ratio was taken from Kohler et al. (2018). The first mixture in the pretest was MC 3 wt% and CNC 1.5 wt% mixed 1:1, resulting in a 2.25 wt% suspension and proved its worth so far and was retained. To better assess the effect of the individual components, the concentration of CNC was increased while the total concentration remained the same. A higher total concentration should also be tested at different ratios of MC and CNC components. The expectation was that if the amount of CNC were increased, the stability to the relative humidity of MC would also increase, because pure CNC should remain stable. It must be mentioned that the 3 wt% CNC showed a very weak adhesive strength in pretests, and therefore a 7 wt% CNC was also included in the test series.

2.2 VISCOSITY

For the application of an adhesive, the viscosity of the fluids is an important issue. To determine the viscosities of the aqueous CNC suspensions, its mixture with aqueous MC solutions and two different MC solutions at different shear rates were measured with a rheometer MCR 300 (table 1).

2.3 TENSILE STRENGTH

The behavior of the selected adhesives and adhesive mixtures (see table 1, only the CNC 3 wt% suspension was removed due to very low adhesion) at different relative humidities was

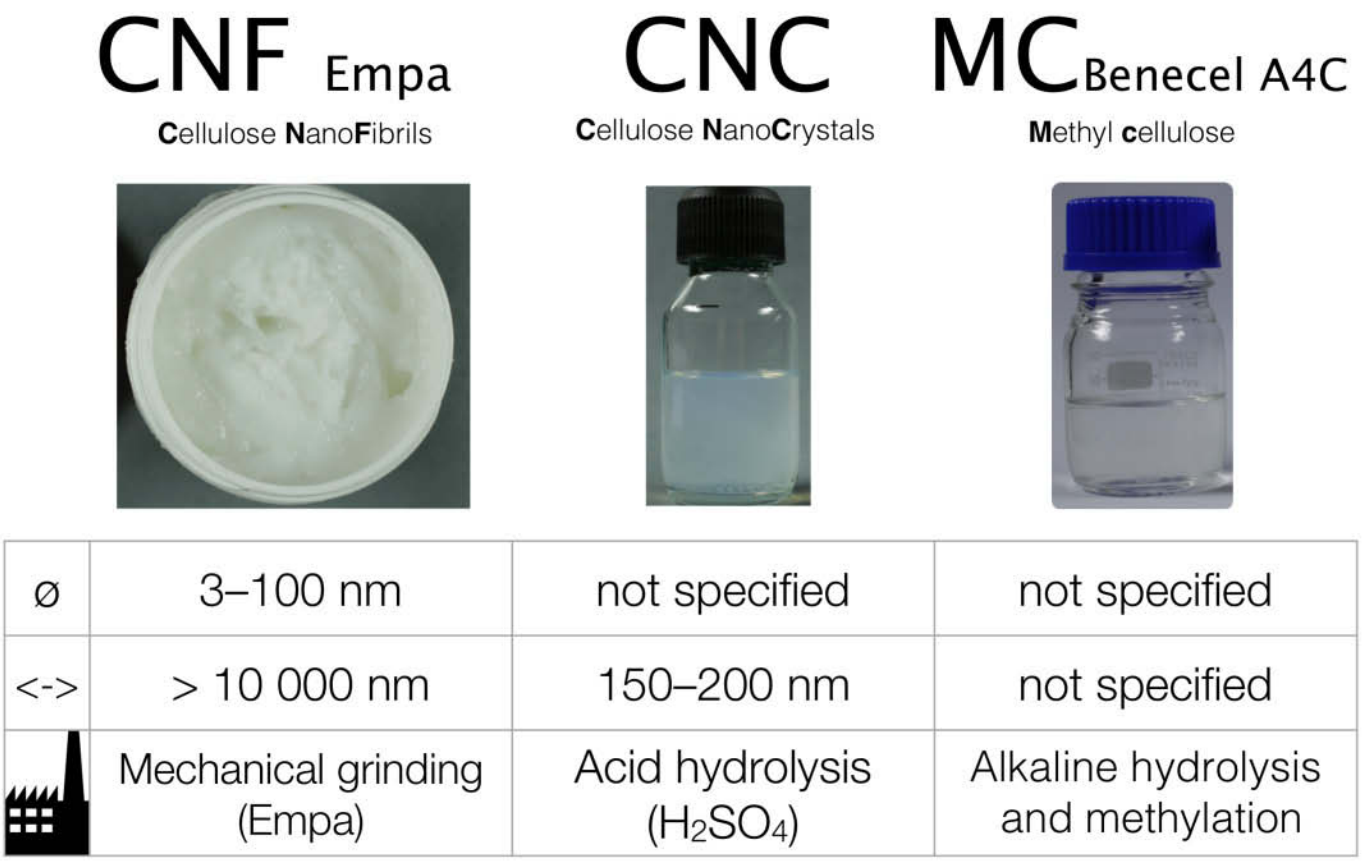

Fig. 2. Suspensions of CNF 3 wt%, CNC 3 wt%, and solution of MC A4C 3 wt%, and their characteristics and production.

Table 1. Measured Solutions and Suspensions

MC A4C wt%	CNC wt%	Ratio	Final wt%
2.25			2.25
3.0			3.0
3.0	1.5	1:1	2.25
1.5	3.0	1:1	2.25
3.0	3.0	1:1	3.0
3.0	3.0	1:3	3.0
	3.0		3.0
	7.0		7.0

determined by means of simple tensile tests. The adhesives were tested on chalk ground flakes (1 cm^2) made of a pigment mixture using chalk, gypsum, and iron oxide red (30:30:1 parts by weight) bound in gelatin, type A, 180 Bloom, 5 wt%. The two chalk ground flakes would imitate failure in the chalk ground of, for instance, wooden sculptures or panels.

The adhesives (7 µg) were applied using a microdispensing device with pressurized air. The accuracy of the applied volume was checked on a precision scale. The samples (1 cm^2) were dried under weight (75 g, 3 × 2 × 1.5 cm) for 7 days. Afterward, they were stored at four different relative humidities (50%, 75%, 84%, 97%) by a constant temperature of 21°C for 1 and 3 months. In the meantime, the flakes were not exposed to any shear or tensile force.

The relative humidity data was, on one hand, taken from an article showing on behalf of MC films how the tensile strength is diminishing by rising relative humidity (Debeaufort and Voilley 1997) and, on the other hand, is based on data taken from churches and castles in Switzerland, Germany, and Sweden (who do not want to be named).

Tensile strength testing was carried out according to Comiotto (2009; see Soppa et al. [2014]) with a Zwick 1120 (Zwick GmbH & Co.; testing speed 50 mm/minute). The results were recorded in newtons per square centimeter and presented with calculated averages and standard deviations. For each test series, 10 samples were tested.

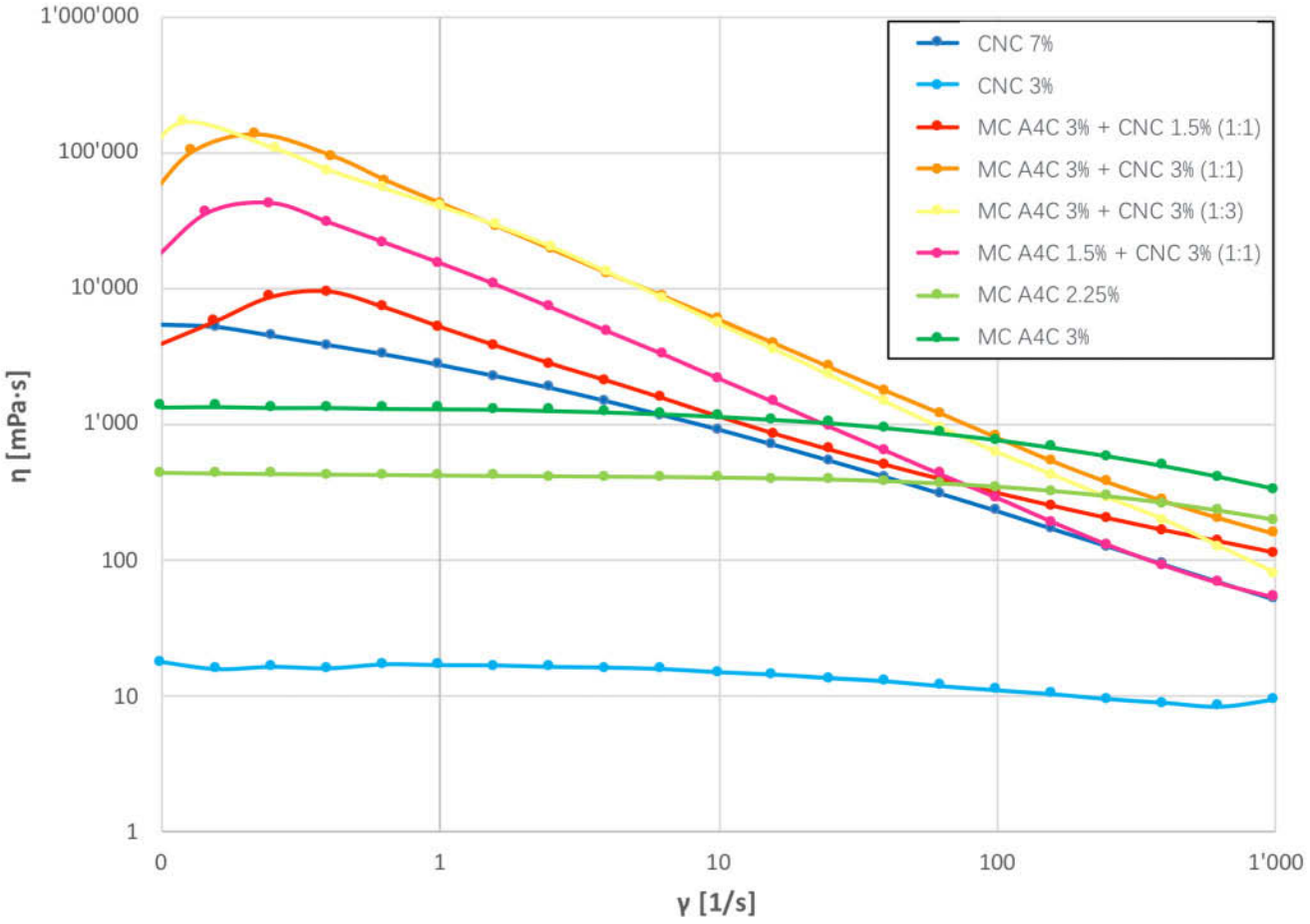

Fig. 3. Viscosity of the suspensions, mixtures, and MC solutions.

3. RESULTS AND DISCUSSION

3.1 VISCOSITY

The aqueous solution of MC at 2.25 wt% and 3 wt% (green lines) show a shear thinning at high shear rates of 10 to 1000 1/second (fig. 3, from left to right). In comparison, a 3 wt% nanocrystalline cellulose (CNC) suspension shows Newtonian behavior, an almost constant viscosity over shear rate. This changes greatly with the 7 wt% CNC, which shows a clear shear thinning.

For the MC-CNC mixtures, the viscosity initially increases before falling, and they all show a stronger shear thinning than CNC 7 wt%. The highest viscosity at rest is shown by the 3 wt% mixtures.

In a nutshell, all mixtures, if stirred, are of lower viscosity than MC 3 wt%. However, the viscosity at rest increases rapidly with the mixtures and CNC 7 wt%. Because CNC 3 wt% are very low viscous and lead to hardly any adhesion of the absorbent chalk ground, a higher concentration of 7 wt% that can still be applied with a microdispensing device was chosen for the tensile strength tests.

3.2 TENSILE STRENGTH

The expectations for the adhesive strength of pure MC at varying relative humidities were based on the results of Debeaufort and Voilley (1997). Here, the ultimate tensile strength of free-standing MC films was determined with 24 N/cm^2 at 50%RH, 11 N/cm^2 at 75%RH, 9 N/cm^2 at 84%RH, and 6 N/cm^2 at 97%RH.

In all results we have large standard deviations, which often overlap. Only at 75% a significant difference between the MC and the mixtures can be seen (fig. 4). Therefore the following differences in adhesive strength are mainly to be understood as tendencies.

After 1 and 3 months adhered chalk ground flakes satisfactorily and, as expected, adhesive strength decreased by more than half at 75%RH. The subsequent increase in adhesive strength was surprising at 84% and 97%RH - written together so far (figs. 4–6). An explanation for this could be an agglomeration of molecular chains, which occurs even in low-percentage solutions (Bodvik et al. 2010 Dogsa et al. 2017) or even the formation of network-forming fibrillar structures of

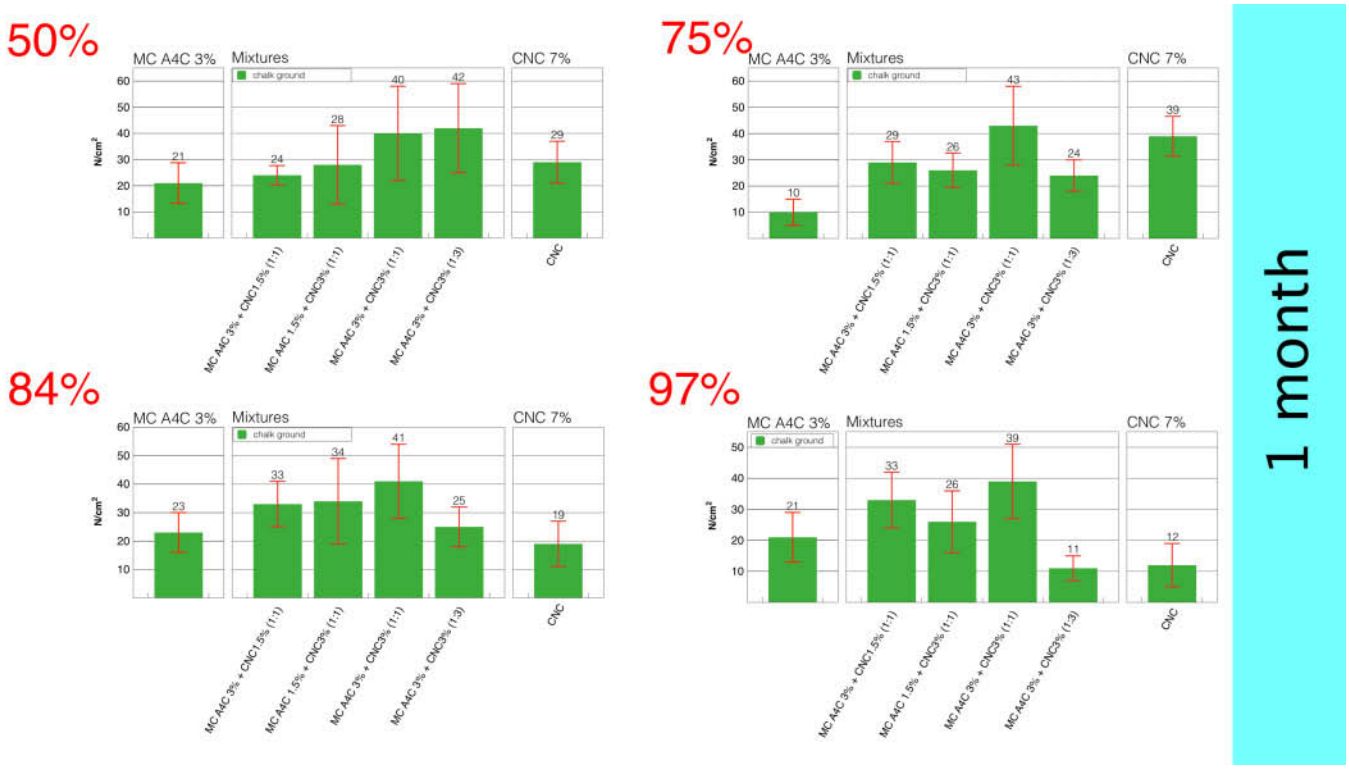

Fig. 4. Tensile strength results after 1 month.

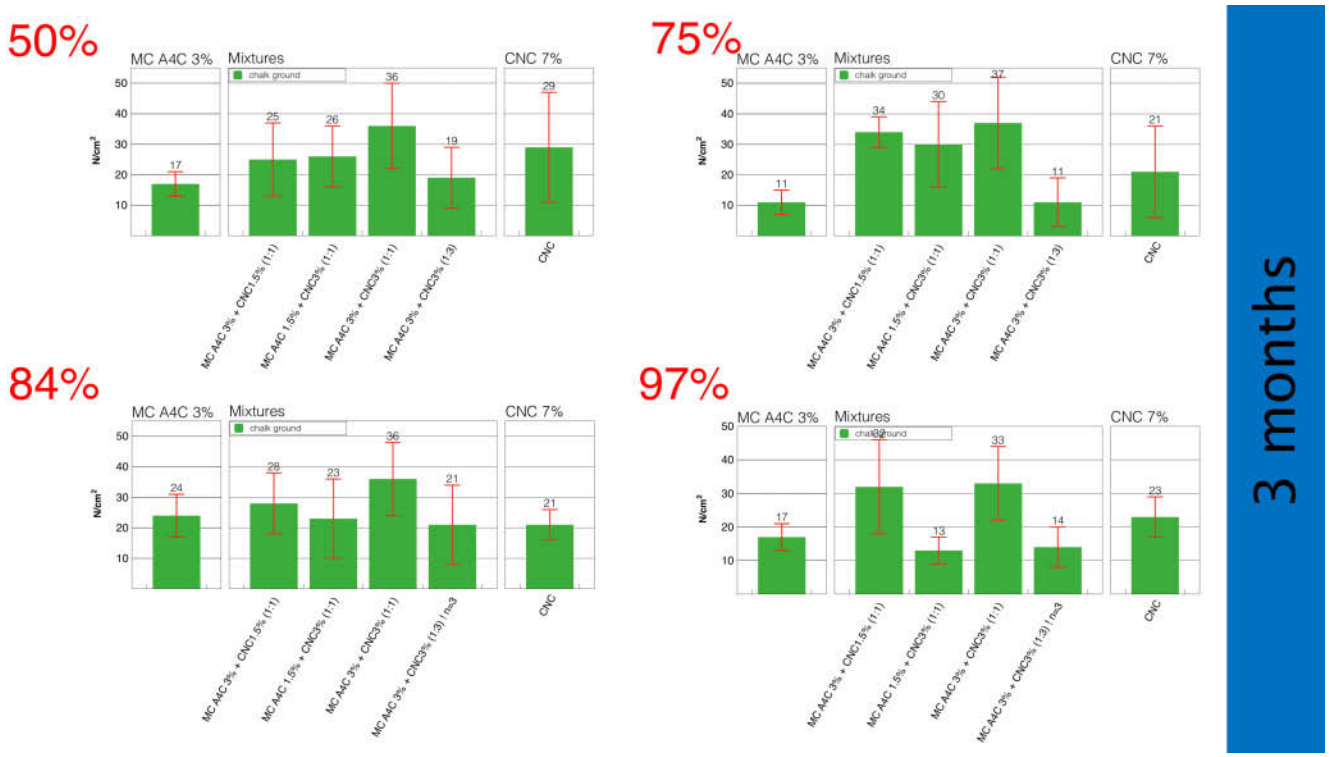

Fig. 5. Tensile strength results after 3 months.

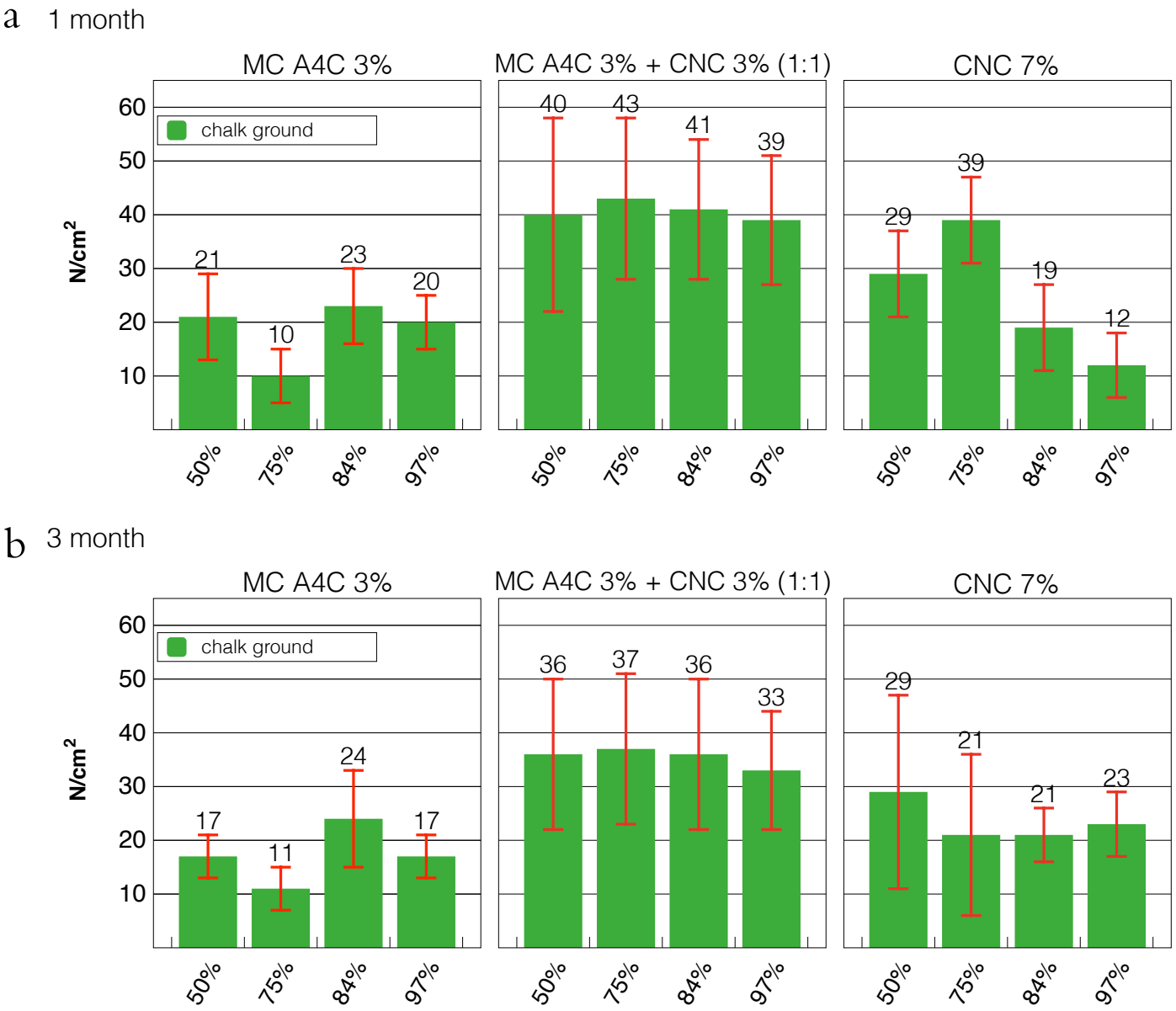

Fig. 6. (a) Selected results of MC A4C 3 wt%, MC A4C 3 wt% + CNC 3 wt% (1:1), and CNC 7 wt% after 1 month. (b) Selected results of MC A4C 3 wt%, MC A4C 3 wt% + CNC 3 wt% (1:1), and CNC 7 wt% after 3 months.

approximately 15±1 nm diameter and approximately 50 nm length of the MC (McAllister et al. 2015a, b) as well as an interaction with the gelatin substrate. McAllister et al. (2015a, b) showed the fibril structure even more precisely and proved that fibrils can form even at 30°C over weeks. Pure nanocrystals initially have a quite similar adhesive strength, which only increases at 75%. Astonishing was the strong decrease of the adhesive strength at 97% up to 13 N/cm^2. This is seen with a layer of water between the CNC crystals and the substrate. The 2.25 wt% MC-CNC mixture, which can be applied very well with a pipette, initially showed only a slight increase in adhesive strength but a satisfactory increase with increasing relative humidity. The 2.25 wt% mixture with more CNC was worse. The 3 wt% (1:1) MC-CNC mixture had the highest and most constant adhesive strength. Therefore, this mixture meets our expectations best (fig. 6).

The loss of adhesion of MC at 75%RH is very noticeable both after 1 month and after 3 months (around 11 ± 4 N/cm^2). Most works of art are exposed to elevated humidity greater than 50% and less than 75%. Especially at 75 RH, the MC shows that the mixtures MC A4C 3 wt% + CNC 1.5 wt% and 3 wt% (1:1) could clearly absorb the

attenuation, as they show significantly stronger ultimate tensile forces (37 ± 9 N/cm^2, 43 ± 14 N/cm^2, 34 ± 5 N/cm^2, 37 ± 14 N/cm^2).

To put the adhesive force into context, we then tested the 25 wt% dispersion medium for consolidation. For 50%RH, the adhesion to the chalk ground was 60 ± 20 N/cm^2 and for 97%RH still 47 ± 9 N/cm^2. However, a substrate fracture almost always occurred, which destroyed the chalk ground. This could not be observed with MC and the mixtures or with CNC. An adhesion fracture close to the substrate almost always occurred.

Surely it is worth mentioning that mold appeared only two times at the side of some chalk ground samples during the 3-month test series at 97%RH. Another notable observation after opening the joints was the color change of the pink chalk ground due to the adhesives: the more CNC, the less color change.

4. CONCLUSION

The results of Debaufort and Voilley (1997) of decreasing cohesion with increasing relative humidity of free methyl cellulose films could not be confirmed here for the adhesion of chalk ground flakes after 1 or 3 months without load at very high humidities.

In all results we have large standard deviations, which often overlap. Only at 75% a significant difference between the MC and the mixtures can be seen. When gluing two chalk layers, the mixture of MC and CNC showed the lowest loss of tensile strength at high humidity, in contrast to pure MC and MC with CNF. These two mixtures of MC and CNC performed best: MC A4C 3 wt% + CNC 3 wt% (1:1) and MC A4C 3 wt% + CNC 1.5 wt% (1:1).

Mixtures with a higher CNC content performed poorly. Whether the adhesive strength or resistance can also be achieved with a lower proportion of CNC should be checked. The adhesive strength achieved is more than sufficient for the bonding of chalk ground. There was always an adhesion break close to the substrate.

Because the mixtures show a shear thinning, it is important to either stir the mixtures permanently or to use a micro-dosing system with compressed air during application. This ensures that the required quantity is adequately distributed under the floe.

This was a first step to approach the situation in castles and churches at high relative humidity. In a further step the areas below 75%RH as well as a longer exposure time or areas around 60-75%RH should be investigated. Future goals include maintaining or even increasing adhesive strength while simultaneously reducing viscosity and minimizing standard deviation as well as tests for transferability to other subtrates.

ACKNOWLEDGMENTS

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SOURCES OF MATERIALS

Saturated Salt Solutions NaCl for 75%RH, KCl for 84%RH, and for 97%RH K_2SO_4

Cellulose nanocrystals CNC, The University of Maine, Process Development Center
5737 Jenness Hall, Orono, ME 04469

Ultra-fine friction grinder "Supermasscolloider"
MKZA10-20J CE, Masuko Sangyo Co., Ltd., Kawaguchi/ Saitama, Japan

Benecel A4C Methyl cellulose
Ashland Inc., USA

Microdispenser, JBE1113 or D3PDSA-02
H. Sigrist & Partner AG, Switzerland, https://shop.dosiersysteme.ch/products/dispensing-and-mixing-technology/dispensing-system/dispensing-system/6591/time-based-analog-dispensing-controller

SpeedMixer, DAC 150.1 FVZ
Hauschild & Co KG, Germany

Precision Weighing Device, Practum224-1S
Sartorius, Germany

Tensile Testing Machine, Zwick 1120 (testing speed 50 mm/minute)
Zwick GmbH & Co., Germany

Rheometer MCR 300
Anton Paar GmbH, Switzerland

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MONICA KATZ

Local Color: The Visual Analysis of a South American Colonial Lacquered Gourd in the Collection of the Hispanic Society Museum and Library

ABSTRACT—The Hispanic Society Museum and Library has a collection of colonial Spanish American lacquered objects, decorated with an indigenous lacquer technique—*barniz de Pasto*. Made for a European aesthetic, mimicking Asian lacquer, the objects demonstrate the craftsmanship of artisans whose techniques are still in use in Colombia today. This study of a gourd in the collection analyzes the decorative elements to identify their sources, showing that artisans regularly substituted local flora and fauna in place of the stylized motifs from Asian lacquer, as well as incorporating designs from European sources. The study also includes considerations on the layered technique, as well as on the pigments and colorants used to create the lustrous effects.

1. INTRODUCTION

The Hispanic Society Museum and Library (HSM&L) was founded by Archer Milton Huntington in 1904, and its collection—always intended to be exhibited to the public—reflected his lifelong passion for all things Iberic. With 35,000 paintings, sculptures, and decorative art objects, as well as more than 250,000 books and manuscripts, the collection spans nearly 4000 years from wherever Spanish and Portuguese were spoken.

The museum is well known for its works by Velazquez and Goya, but among the more intriguing objects in the collection are a small group of South American colonial lacquers, acquired largely in the past 30 years, made by indigenous artisans for a European aesthetic and meant to imitate Asian lacquers. Although the technique has survived to this day, there is scant documentary evidence describing the evolution and manufacture of these beautiful and intricately decorated objects—so sophisticated was their decoration that they have often been mistakenly identified as the Asian lacquer objects they sought to imitate.

The subject of this study is a lacquered gourd that entered the Hispanic Society collection in 2014 (fig. 1). Made from a bottle gourd (*Lagenaria siceraria*) or possibly a totuma (*Crescentia cujete*), in Pasto, Colombia, it is decorated with *barniz de Pasto* and silver leaf.

Barniz de Pasto describes an indigenous waterproofing and decorative technique that uses a translucent pale green resin produced by *Elaeagia pastoensis Mora*, a plant native to tropical rainforests in the upper Andes and commonly known as mopa mopa. In terms of geographical range, the mopa mopa tree grows as far north as Costa Rica and Panama and as far south as Peru and is one of two verified species of *Elaeagia* known to produce resin used in this way. In 2015, Richard Newman at the Museum of Fine Arts, Boston found a way to differentiate between the two; he confirmed that the resin used on the Peruvian qero cups, visually different and decorated with a different method but previously thought to also come from *Elaeagia pastoensis Mora*, was in fact from the related species, *Elaeagia utilis* (Newman 2015).

The inspiration for this article was the theory of Dr. Mitchell Codding, director of the HSM&L, who had found local-seeming flora and fauna replacing typically Asian decorative elements in the design of a ca. 1684 *barniz de Pasto* box in the HSM&L's collection—in other words, local color on one of the world's first global objects (Codding 2006, 2015). The examination of the lacquered gourd was particularly interesting in this context, because unlike the other *barniz de Pasto* pieces in the collection that are all boxes, a form introduced by the Spanish, the gourd is an indigenous form, only 12 cm high and 11 cm in diameter.

2. STUDY PARAMETERS: VISUAL ANALYSIS WITH AVAILABLE EQUIPMENT

To obtain clear images of the tiny decorative details, images were taken with a Nikon SMZ 900 stereoscopic zoom microscope; other images were obtained using an iPhone XS Max with all three Olloclip macro lenses (x7, x14, x21) and then compared with images of flora and fauna in recently published field guides for the region cognizant of the impossibility of quantifying changes in biodiversity from the 17th century.

3. TECHNIQUE

Mopa mopa resin is the plant's protective coating for its young leaf shoots. It was collected, cleaned of plant debris by boiling, and then chewed to soften it and regain elasticity before being stretched using teeth and hands into onionskin-thin sheets. Decorative elements were cut out of the thinnest central part of these sheets and applied, with heat, directly to the substrate. The elasticity of the resin is such that a 2013 paper by engineers at

Fig. 1. Gourd, Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Fig. 2. Gourd (flower detail), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

San Buenaventura University in Cali, Colombia, studied its possible use as a polyethylene substitute from a renewable source (Toro, Mina, and Bolaños 2013).

Heat is central to the fabrication of *barniz de Pasto* objects, and artisans therefore had to work quickly, making the intricacy of the designs all the more remarkable and the randomness of details less likely. The flower in figure 2 measures only 15 mm across. On top of the base layer of resin—naturally a greenish-yellow—is a sheet of silver leaf, covered with a green-tinted sheet of resin. The shape of each of the four larger petals is cut out of another sandwich of silver leaf between resin, the piece of red-tinted resin on top, and adhered to the green resin. Evidence of this can be seen clearly in figures 2 and 3. In figure 2, note the textural difference between the foil under the green resin and that under the red resin, and the black line marking the join of two pieces of silver leaf above the whole flower. In figure 3, the edge of the red-covered silver leaf is clearly visible just below the cream outline of the petal on the left-hand side. The petals' opaque cream outline is inlaid. Also inlaid are the shapes of the inner petals and each individual filament—there are up to 19 per petal, measuring only 2 mm long and no more than 0.15 mm wide. The shapes for the stigma at the center and the four sepals, as well as the four tiny

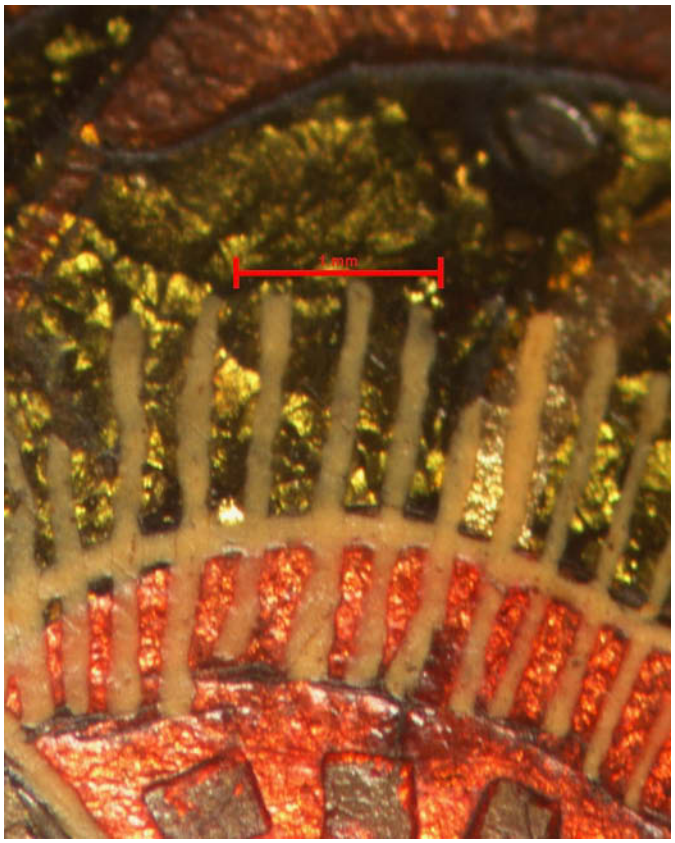

Fig. 3. Gourd (flower detail with scale), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Fig. 4. Coffer, Pasto, Colombia, ca. 1650. Wood with *barniz de Pasto*. 15 x 18 x 8.6 cm. Hispanic Society of America, New York. LS2361.

squares in each petal, were each excised, and untinted pieces of resin were inlaid, revealing the metal leaf below. Now oxidized, these tiny squares originally would have appeared as silver.

4. DATING THEORY

The gourd has an approximate date of 1675. In the colonial period, two different techniques using *Elaeagia pastoensis Mora* developed contemporaneously: the *barniz brillante* of this gourd and objects decorated with pigment-saturated resin, *matte barniz*, like the ca. 1650 box in figure 4. Based on comparisons with other similarly decorated objects, and absent any documentary evidence, the theory is that the more sophisticated the work and intricate the design, the closer to the source of the tradition. Earlier pieces like the gourd incorporate iconography found in medieval illustrated manuscripts. Later pieces tend to include more generic and simpler flora and fauna and are more imitative of Asian lacquer. The ca. 1684 writing box that inspired this study has replaced the more typical squirrels in Asian lacquer with monkeys and included Andean berries—*agraz*—instead of grapes (fig. 5). One of the motivating forces in this research is the hope that by examining the decorative elements more closely, they will provide clues to a more accurate timeline for these objects.

Fig. 5. Portable writing desk, Pasto, Colombia, ca. 1684. Wood with *barniz de Pasto*, yellow metal hardware. 20 x 31 x 36 cm. Hispanic Society of America, New York. LS2000.

5. DESCRIPTION AND SOURCE OF THE GOURD'S DECORATION

The gourd's decoration is divided into four panels: three rectangular panels each framed with a red border with gold-colored crescents and one circular panel at the base (figs. 6–9). At the center of each panel is an animal (a griffin, a unicorn, a large cat, and a deer). All backgrounds are crammed mainly with flora but also several types of birds that appear in each of the four corners of the three rectangular panels.

The unicorn and the griffin in figures 6 and 7 have clear European roots, and both were used frequently to illustrate medieval Books of Hours and other liturgical or secular texts (Codding and O'Neill 2006). The unicorn is a mythical beast—a symbol of purity—and has long been associated with the worship of the Virgin Mary. The griffin symbolizes the dual nature of Christ: the divine represented by its eagle's head, wings, and talons and the human symbolized by the animal (the lion's legs) (Hall 1979).

The source of the cat in figure 8 is a little less familiar. Large cats appear frequently in both European and South American art. They were important to indigenous cultures, and several large felines were native to Colombia. Originally thought to be representative of a jaguar, with inlaid spots of a different-colored resin, the incredibly bushy and detailed tail might indicate a jaguarundi or a coati, an indigenous member of the raccoon family (McMullan and Donegan 2014).

The deer in figure 9, also common to both European and South American art, appears frequently in European manuscripts, but there are also deer native to Colombia, such as the red brocket deer (*Mazama americana*) (McMullan and Donegan 2014).

The birds depicted on this gourd are of a few different types. More likely from European sources are the peacock on the cat panel shown in figure 8 (a symbol of immortality) and a pair of owls on the unicorn panel in figure 6 (a symbol of wisdom) (Hall 1979), which closely resemble the mottled owl (*Ciccaba virgata*) found in the Colombian department of Nariño, which has Pasto

Fig. 6. Gourd (detail of griffin panel), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

as its capital (McMullan and Donegan 2014) and may be the source.

And what of the smaller birds and the floral elements? Here the artisans might have had more freedom of expression, limited only by the medium and the narrow palette of this object. There are several types of birds on this gourd, mostly rendered in a combination of white and pink, with one in gold. Colombia has more than 1800 confirmed species of birds, and a search in the field guide using the birds' most basic physical characteristics (their size and whether they had long tails or crests) yielded very few feasible matches to the three basic types depicted on the gourd, even when it became clear that the birds' colors were fictitious. Most

Fig. 7. Gourd (detail of unicorn panel), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Fig. 8. Gourd (detail of cat panel), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

of them, especially the majority of small white birds, appeared more like the generic birds found in medieval manuscripts (Codding and O'Neill 2006; McMullan and Donegan 2014).

Identification of the flora was equally complicated: Dr. Francisco Morales, a Colombian ethnobotanist, commented that the plants are "100% chimeric. The artist drew flowers and inflorescences from different species in a single plant, and they

Fig. 9. Gourd (detail of deer panel), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Fig. 10. Gourd (detail of passion fruit bud), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

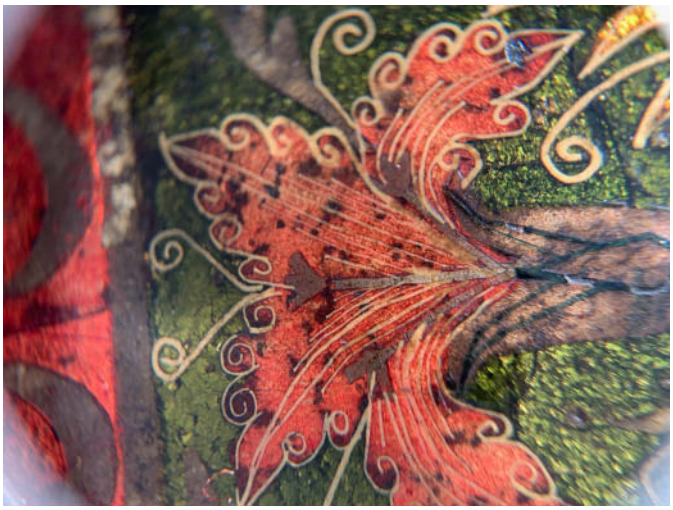

Fig. 12. Gourd (detail of orchid), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

look mostly ornamental" (Morales, pers. comm.). Nonetheless, it was possible to identify plants that would have grown locally and resemble decorative elements on the gourd (figs. 10–13), such as passion fruit (*Passiflora*) flower buds in figure 10 and passion fruit flower in bloom in figure 11, orchids (e.g., *cattleya trianae*, the present-day national flower of Colombia) in figure 12, and annatto (*Bixa orellana*), agraz, or Andean berries in figure 13 (*Vaccinium meridionale*), which are quite distinctive and appear in all the *barniz de Pasto* objects in the HSM&L collection (Gentry 1996).

Inspiration for the geometric designs in the two border areas (figs. 14, 15)—gold triangular motifs on the blue inner rim in figure 14 and gold crescents on a red background bordering each panel in figure 15—came both from the objects' indigenous origins and from being an Asian influence. Triangles appear everywhere in pre-Colombian art and on the gourd are reminiscent of the Inca step design (Botero 2008).

However, the crescent design was harder to identify. Many of the *barniz de Pasto* objects have a border with some kind of repeating design but not with overlapping crescents, which was not common pre-Colombian decoration. The use of a border to create "compartments" or panels was a common device in European art but also in the Namban lacquer chests that were imported in Spain throughout this period (Kawamura 2018). Dr. Morales further suggested that they may represent seed pods (Morales, pers. comm.).

Fig. 11. Gourd (detail of passion fruit flower bloom), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Fig. 13. Gourd (detail of Andean berries), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

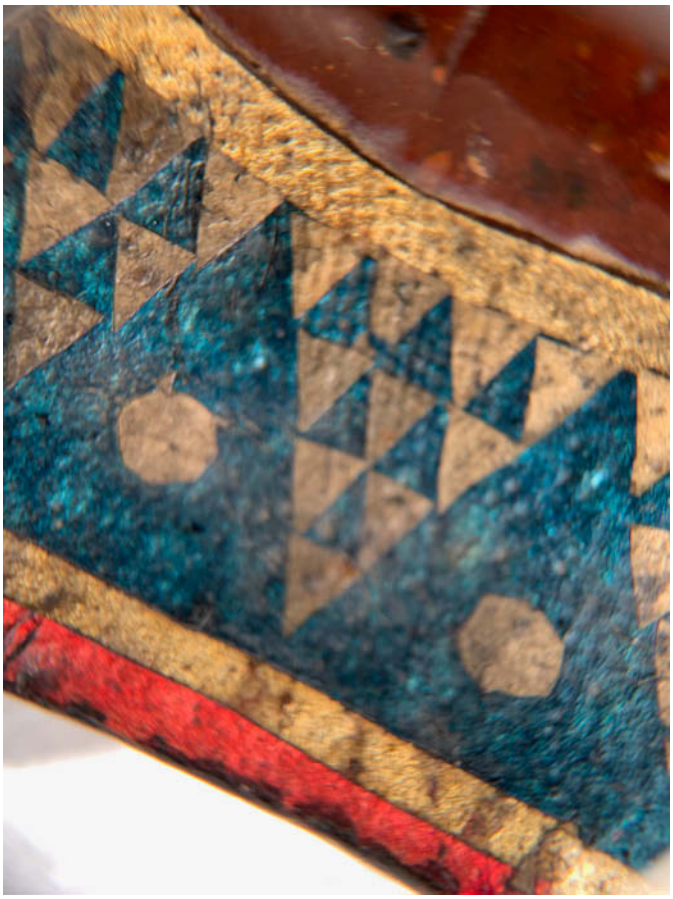

Fig. 14. Gourd (detail of blue border), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

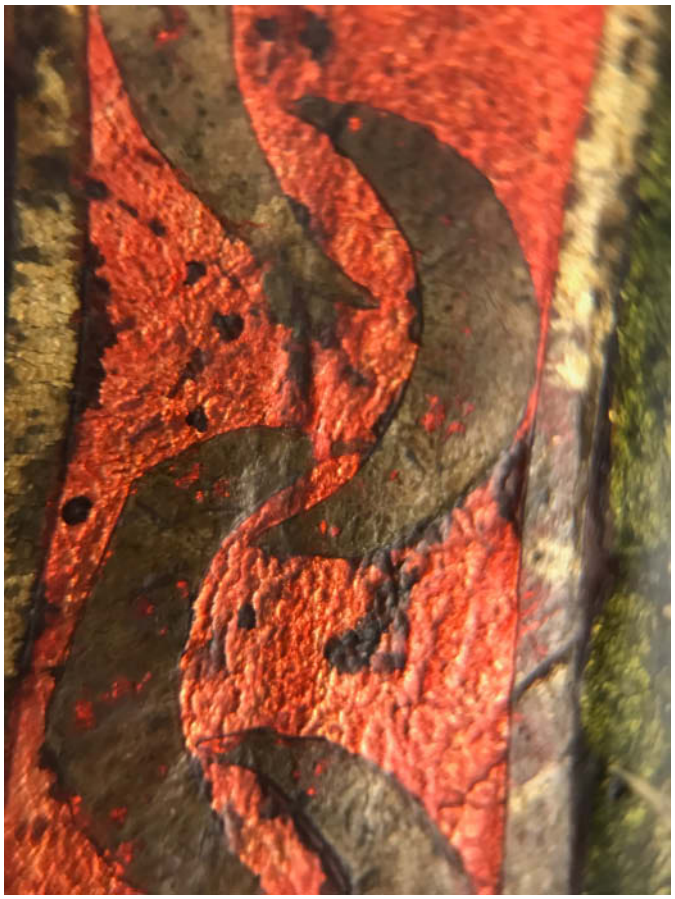

Fig. 15. Gourd (detail of red border), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

6. PIGMENTS, COLORANTS, AND OTHER FINDINGS

The gourd has a limited palette of translucent colors over metal leaf: the green background; red, yellow, pink, saturated white, cream, and black used to outline the decorative elements; and blue and gold. Writing in 1801 of his experiences traveling through the region, Alexander von Humboldt, the German naturalist and explorer, identified the colorants used in *barniz de Pasto* objects as dilute indigo for blue; pure indigo for black; annatto for red; the powdered *Escobedia scabrifolia*, a saffron-like root over silver leaf, for gold; and lead oxide for white (Codding 2015). These mostly organic pigments were available locally: Was this also "local color"?

Initial testing using imaging techniques has been performed noninvasively as part of the Metropolitan Museum's Network Initiative for Conservation Science. A comprehensive materials study on this colonial *barniz de Pasto* gourd is currently being carried out through an array of portable and benchtop instruments, including spectroscopic and chromatographic techniques, to identify the pigments and colorants present and provide a detailed characterization of the resin. Radiocarbon dating and wood identification will complete the technical information on the object.

Fig. 16. Gourd (detail of owl showing black as indigo), Pasto, Colombia, ca. 1675. Gourd with *barniz de Pasto*. 12 x 11 cm. Hispanic Society of America, New York. LS2400.

Images collected under magnification have already confirmed that the dark outlines that characterize some of the inlayed decorations, which appear black to the naked eye, are instead made with a dark blue–tinted resin as can be seen in figure 16, in which the owls' detailing looks green. Observations at the stereomicroscope also suggested the possible use of two silver leaves in the decoration, as seen in figures 2 and 3. The ongoing scientific study will help confirm the presence of indigo, hypothesized as the source for the blue colorant, and will determine the exact composition of the silver leaf.

An important new consideration will be the 2018 discovery that calomel ("mercury white") had been used as a pigment on a *barniz de Pasto* box in the Victoria and Albert Museum's collection (Burgio et al. 2018). Reviewing the results from other piecemeal analysis on HSM&L objects in the past, XRF readings on two 17th-century coffers had also shown surprising mercury peaks.

The spikes of mercury in many of the areas tested were not initially surprising because it was assumed that the mercury levels were a by-product of colonial methods of silver extraction. The Patio process had been introduced to the Americas in 1571 and used mercury amalgamation to extract silver from the lower-grade ores mined, for example, famously in Potosi, Bolivia (Brown 2012). The department of Nariño even had its own silver mines: there had been a mine producing silver in Mallama, just over 100 km from Pasto (Miller and Singewald 1919). Initially in the 16th century, mercury used in the Patio process was imported from Spain, but there also were mercury deposits in South America. In addition to the famous mercury deposits in Huancavilca, Peru, discovered in the 16th century (Brown 2012), a 2011 Colombian government survey noted seven occurrences of cinnabar in Nariño (Brooks 2014).

Clearly, the use of indigenous materials by local artisans must have given South American lacquers some particularly local inspiration for their decorations, but these are as yet not quantifiable. The initial study of this particular object was facilitated by relatively cheap and readily available lenses for a smart phone. Noninvasive imaging techniques were able to characterize some of the pigments used, but further sampling currently under way will be necessary to fully identify the materials used on this beautifully decorated and unusual object.

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MOHAMED MOUSTAFA*, IBRAHIM EL-RIFAI, MOHAMMED S. A. KHEDR, AND NAGLAA MAHMOUD

Archaeometric Study of a Gilded Wooden Statue from the Ottoman Period

ABSTRACT—The statue studied here of a servant African woman was found before an attempt to smuggle it out of Egypt. It is likely that it dates back to the period of Muhammad Ali and his family. This statue is made of gilded wood with a black face and decorated with multiple colors on the gilded layer. In addition, it is designed to rotate 360 degrees. Here we will shed light on identifying the botanical species of wood and the chemical composition of the materials used decoratively. Visual assessment, optical microscopy, multispectral imaging, SEM-EDAX, XRD, and FTIR spectroscopy were used in this study to elucidate the components of this statue. The microscopic observation of wood thin sections from several places made it possible to identify pine wood as the wood used in this statue. The analyses provided offer more information concerning the original materials and those added during the previous treatment interventions, which need to be considered when applying a future conservation plan.

1. INTRODUCTION

Materials analysis has long been recognized as an essential component of archaeological research. It yields critical compositional information that may lead to object characterization, revealing ingredients and technologies used by craftsmen (Ciliberto and Spoto 2000; Uda 2005; Price and Burton 2011). Furthermore, in the context of object conservation, it allows informed decisions necessary for proper conservation/restoration of objects to be made (Stuart 2007). The statue studied here of a servant African woman was stolen but found before an attempt to smuggle it out of Egypt. It is likely that it dates back to the period of Mohamed Ali and his family. The statue mainly consists of two pieces. The body has a black face and is decorated with multiple colors on the clothes and base, which was designed to rotate the body of the statue 360 degrees. The main component is wood covered with a prepared ground layer, then a gilded layer; multiple colors were applied on the gilded layer. The statue is about 200 cm in length and 65 cm in width. This study aims to combine SEM-EDAX and multispectral imaging (MSI) to identify the original pigments, and previous conservation interventions used in decorated areas, also using XRD and FTIR–attenuated total reflection to identify the ground layer, binding media, and varnish covering the whole statue. Moreover, identification of wood species is included in this study.

2. MATERIALS AND METHODS

Scientific analytical techniques, such as optical microscopy (OM), an environmental scanning electron microscope (ESEM) supported with EDAX, XRD, and FTIR spectroscopy, and MSI were applied to elucidate the nature of the original and added materials, explain the deterioration aspects, and establish the proposal of conservation for the statue.

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2.1 VISUAL ASSESSMENT

Visual assessment, by the critical eye of our team, was performed to determine the deterioration aspects of the gilded statue. This method is very effective because the causes and deterioration aspects may be easily identifiable. The critical eye of a conservator can also determine the most effective techniques of analysis to be applied for identifying the condition of the statue under study (Abdel-Maksoud 2011; Lo Monaco et al. 2013).

2.2 OPTICAL MICROSCOPY

For the transmitted light microscope, OPTIKA microscopy (Italy) equipped with an OPTIKA B9 digital camera was used to identify the wood species. Thin sections were obtained in the three principal anatomical directions: transverse, tangential, and radial. The observation and description of the anatomical features of the sample were based on wood anatomy atlases (Ismail, Abdrabou, and Abdallah 2016), whereas a USB digital microscope with magnification ratio 1000x was used to get detailed photos of pigments and obtain more information about the sequence of layers.

2.3 MSI TECHNIQUE

MSI has long been used in the field of artwork investigation. The infrared (IR) region constructs an important part in this regard, as it has many applications such as underdrawings inspection and pigment identification (Verhoeven 2008). In this study, visible (VIS), visible-induced UV luminescence (UVL),

visible-induced IR luminescence (VIL), and IR were acquired with a FujiFilm S5 Pro modified camera for "full spectrum" (between 380 and 1000 nm). The camera was calibrated with the X-rite Color Checker Passport and its bundled software to create a camera profile for Adobe Camera Raw. The images were shot RAW and were then color corrected, using the camera profile, and white balanced (Verri et al. 2008; Dyer, O'Connell, and Simpson 2014). For visible imaging, the excitation was provided by two photographic white light fluorescent sources and the camera lens was fitted with a B+W 486 UV-IR-CUT filter (88% cut of UV at 380 nm, 95% cut of IR at 700 nm). For IR imaging, the excitation was provided by 75W IR reflector light, and the camera lens was fitted with B+W IR filter 093 (1% transmission at 800 nm to 88% at 900 nm). In addition, the UV imaging was provided by an 18W UV black light (approximately 365–395 nm), and the lens was fitted with B+W UVA filter 403 (320–385 nm) (El-Rifai et al. 2013; El-Rifai, Mahgoub, and Ide-Ektessabi 2016).

2.4 ESEM supported with EDX

The Philips XL-30 ESEM in the laboratory of the nuclear materials authority was applied for mineralogical characterization. The analytical conditions were 30 Kv. Accelerating voltages were 1- to 2-mm beam diameter and 60- to 120-second counting times. Minimum detectable weight concentration was from 0.1 to 1 wt%. Separated minerals were picked under a binocular microscope and identified using ESEM supported by EDX 2.4 (Raslan and Fawzy 2018) (Table 1).

2.5 X-ray diffraction

XRD is an analytical technique in which a prepared sample is bombarded with an x-ray beam at varying angles. The diffractometer measures the size of the "unit cell" (the space between adjacent crystal planes) by virtue of the d-spacing. x-ray diffraction using the x-ray diffractmeter system PW3040-analytical equipment-PANalytical Pro model, and a Cu target tube and Ni filter at 40 kv and 30 MA, with X'pert High score software, were used for identifying the components of the ground layer (Moustafa et al. 2018).

2.6 FTIR–attenuated total reflection

FTIR spectroscopy measurements were performed using an FTIR spectrometer (Vertex 70, Bruker) equipped with an attenuated total reflection accessory, in the 400 to 4000 cm^{-1} range, with spectral resolution of 8 cm^{-1}. The organic materials were identified by comparison of the obtained spectra with data from the literature (Derrik, Stulik, and Landy 1999) and reference spectra obtained in the laboratory.

Table 1. EDAX Results of the Painted Layers and Previous Conservation Materials

Elements	Previous Conservation Materials on Yellowish Areas (%)	Black Painted Layer (%)	Previous Conservation Materials on Black Areas (%)	Green Painted Layer (%)	White Painted Layer (%)	Red Painted Layer (%)	Blue Painted Layer (%)
C	3.16	43.12	39.32	–	52.13	–	20.39
Ca	8.20	31.44	28.12	2.66	40.78	4.67	17.29
S	1.89	20.78	–	3.49	3.12	14.52	15.05
Cd	63.29	–	–	–	–	–	–
O	6.40	1.78	5.48	6.28	2.20	–	–
Ti	–	–	25.10	–	–	–	0.75
Pb	–	–	–	–	–	–	–
Hg	–	–	–	–	–	32.13	–
Si	3.89	–	–	2.12	1.05	25.56	1.12
Al	3.34	–	–	1.62	–	17.89	0.95
Mg	–	–	–	–	–	1.25	–
Cu	2.75	–	2.20	79.65	–	–	–
Cl	5.48	2.88	–	4.18	–	–	–
Fe	1.60	–	–	–	–	2.39	–
K	1.60	–	–	–	–	1.59	–

Fig. 1. (a–c) Examination under a USB microscope.

3. RESULTS AND DISCUSSION

3.1 EXAMINATION BY OPTICAL MICROSCOPE

3.1.1 USB Microscope

Light microscopy is an analytical tool that can provide unique information on a wide range of historical materials. This data can be used for a huge number of purposes, from identifying the history of an artifact to providing insights into the preservation method used. Historical artifacts, in general, have high heterogeneity, which means that information hidden from the naked eye can be obtained by analyzing their microstructure (Osman, Zidan, and Kamal 2014). Using a USB microscope provided more information regarding the sequence of layers, which proved that the main component was wood covered with a gesso layer and a gilded layer, and that the painted layer was applied on the gilded layer (figs. 1a, 1b). In addition, the artist carved all decorations on the ground layer and then applied the gilded layer (fig. 1c).

3.1.2 Identification of Wooden Species

OM examination of the three directions of the wood species used in making the wooden statue revealed the presence of pine wood (*Pinus* spp.). Pine wood was brought from Syria in ancient Egypt (Sharma et al. 2015), and there is a lot of evidence for the use of pine wood in Qubtic and Islamic artifacts. The anatomical characteristics of *Pinus* spp. by OM in transmitted light, transverse section (fig. 2a), showed that growth ring boundaries were distinct. Latewood tracheid thick-walled axial intracellular resin canals were present, with epithelial cells being thin walled. The tangential section (fig. 2b) showed an average ray height medium (5–15 cells). Rays exclusively were uniseriate. Radial intercellular resin canals were present. Radial section (fig. 2c) showed that pitting in radial walls of early wood tracheids predominantly was uniseriate. Ray tracheids commonly were present. Cell walls of ray tracheids were dentate. End walls of ray parenchyma cells were smooth. Horizontal walls of ray parenchyma cells were smooth (unpitted). There was cross-field pitting fenestriform ("window like"). There were one to two bits per cross-field in

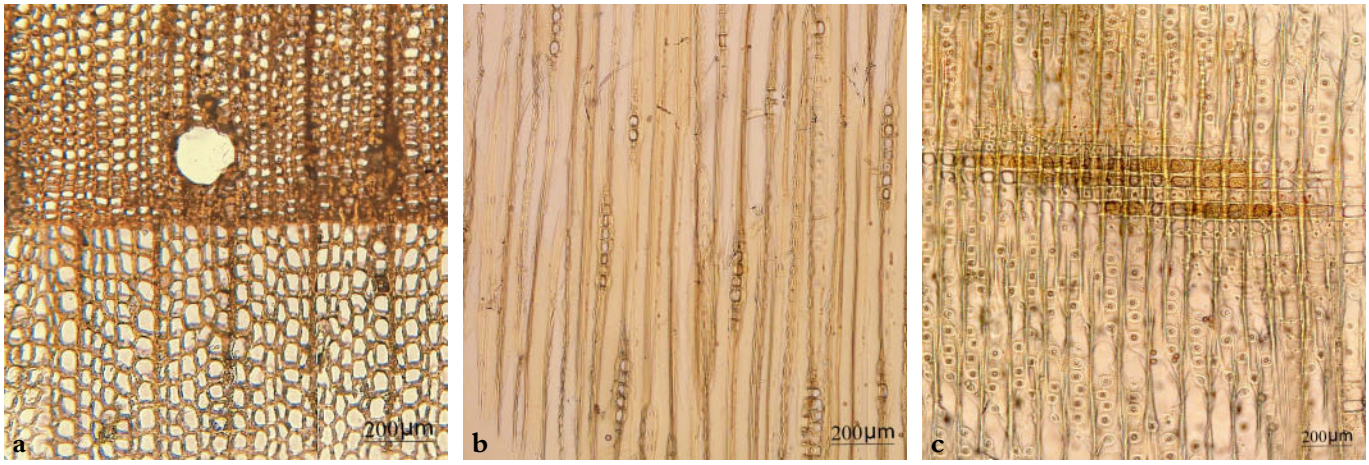

Fig. 2. (a) Transverse section. (b) Tangential section. (c) Radial section.

early wood (large fenestriform). Prismatic crystals located in cells were associated with intercellular canals (Crivellaro and Schweingruber 2013).

3.2 IDENTIFICATION OF THE GROUND LAYER

3.2.1 Ground Layer

XRD results indicated that the ground layer was made out of calcium sulfate (CaSO4) (fig. 3), whereas the calcium carbonate was absent. In addition, the IR spectra of the ground layer sample showed the presence of typical vibration bands of calcium sulphate hydrate, commonly called *gypsum* (CaSO4 × 2H2O), centered at 1103, 667, and 596 cm^{-1}, as well as the stretching and deformation vibrations of the O-H bond of water at 3508, 3397, and 1681 cm^{-1}, respectively (La Russa et al 2009), and bands assigned specifically to animal glue at 1537 and 1619 cm^{-1} (fig. 4) (Glavcheva et al. 2014).

3.2.2 Identification of the Previous Conservation Interventions

The painted polychrome statue was restored previously. MSI was used to elucidate the presence of previous conservation areas, which were confined to the gilded layer and the black painted layer. In addition, MSI gave preliminary indicative results regarding the kind of pigments and previous conservation materials, whereas ESEM supported with EDAX was used for identifying the elements of the previous conservation materials and proved the primary results of MSI (Schreiner, Melcher, and Uhlir 2006). A UV image of the statue face indicated the presence of retouching areas by using black pigment, whereas the original layers were covered with varnish (figs. 5a, 5b). ESEM supported with EDAX of the original layer revealed the presence of carbon (C) in high concentration, with suggestion of using carbon-based pigment in the black areas (fig. 6). The results of previous conservation retouching revealed the presence of titanium (Ti), whereas the carbon was absent, suggesting the use of titanium-based pigments in retouching of the black areas (fig. 7). FTIR results of varnish revealed the presence of absorption bands at 3396 and 3497 cm^{-1} revealed the hydroxyl group (O–H) stretch band, whereas bands at 2850 and 2918 cm^{-1} revealed the alkanes (C–H) stretch band. The absorption band at 1705 cm^{-1} indicated the presence of the sat ketone, acid group (C=O) stretch band. In addition, the absorption band at 1462 cm^{-1} revealed the presence of the alkanes group (CH2) band. All of these results gave a strong suggestion of using shellac as varnish (Martin-Ramos et al. 2018) (fig. 8). The processing of the multispectral images of the yellowish area did not show a clear photo without a polarizing filter because of covering the whole statue by a shiny varnish (fig. 9a), whereas after using the filter, it became clear that the areas of previous conservation interventions were found (fig. 9b). It showed reddish luminescence of the pigment in the UV-induced visible image (UVL) (figs. 9c, 10 a,b). It had a dark shade in the UV-reflected image (UVR) (fig. 10c) and a bright shade in the IR-reflected image (IRR) (fig. 10d), whereas it showed a white color in the IR–false color image (IRFC) (fig. 10e). This suggested that the pigment could be cadmium yellow (Cosentino 2014). ESEM supported with EDAX proved the results from MSI and revealed the presence of a high concentration of cadmium (Cd), the main

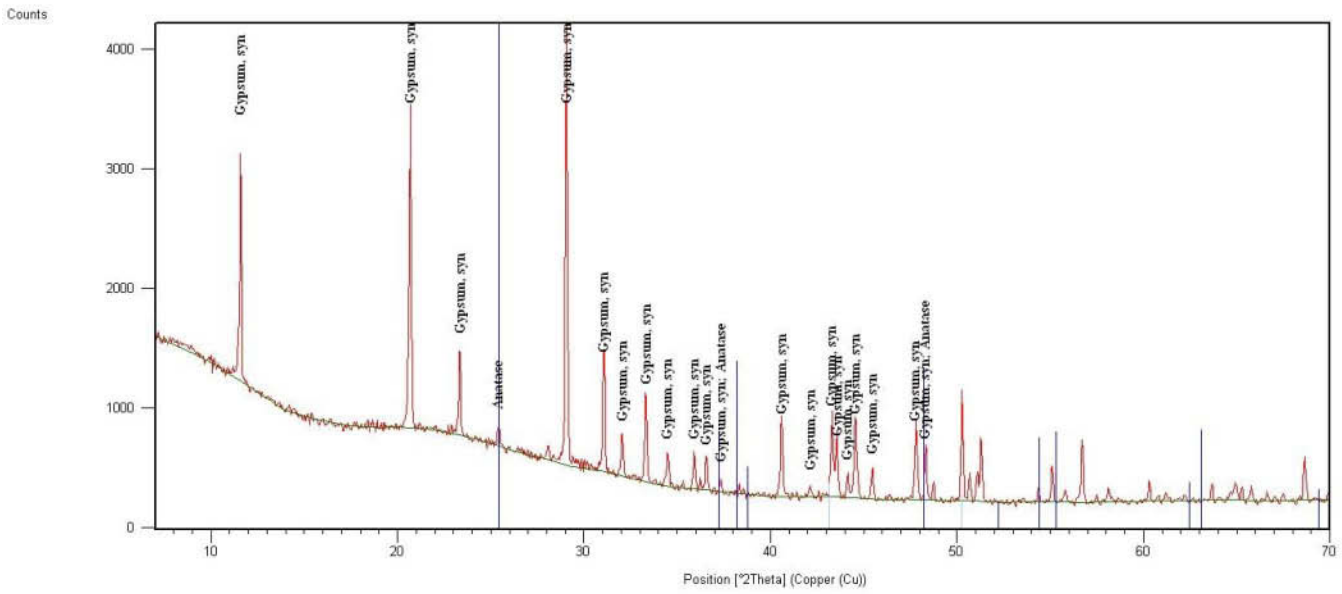

Fig. 3. XRD results of the ground layer.

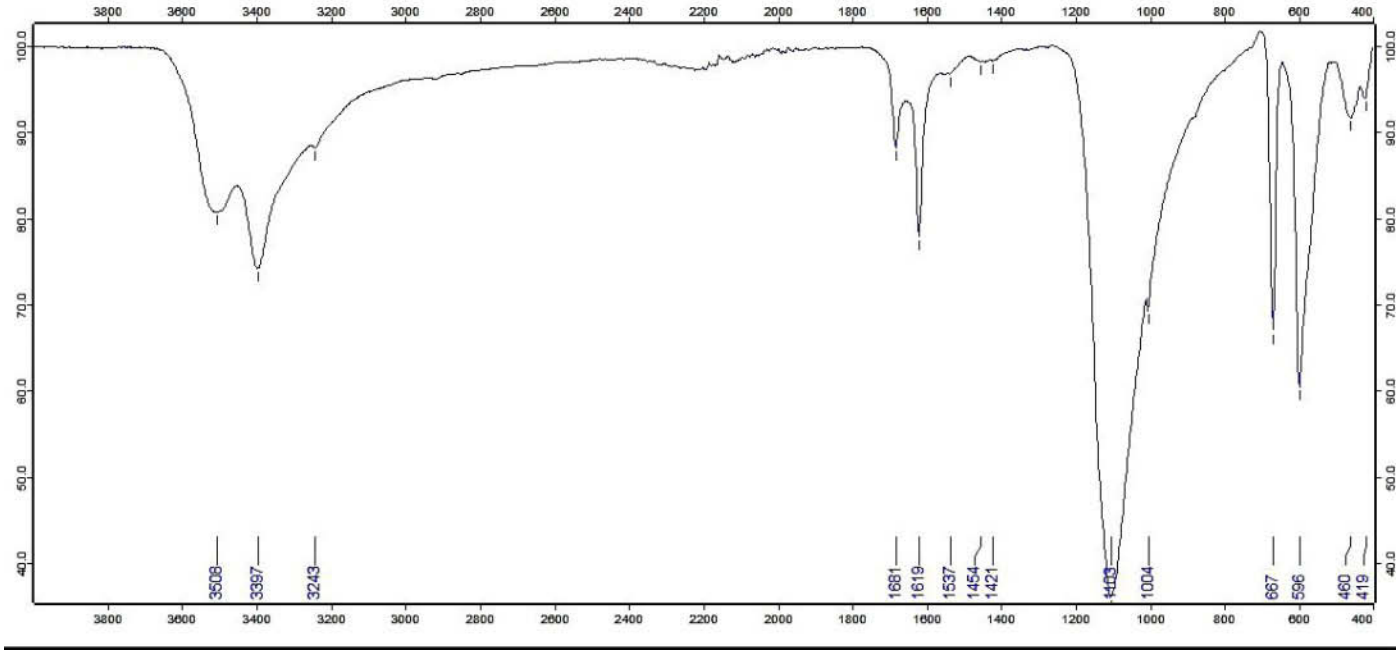

Fig. 4. FTIR results of the ground layer.

component of cadmium yellow (CdS), and sulfur (S), whereas arsenic (As) was absent, which meant that cadmium yellow was used in retouching the missing parts of the gilded layer (fig. 11) (Sekhar, Kumar, and Rao 2015).

3.3 IDENTIFICATION OF THE GREEN PIGMENT

The processing of the multispectral images of the green pigment (fig12a) showed no luminescence of the pigment in the UV-induced visible image (UVL) (fig. 12b). It had a dark shade

Fig. 5. (a) Visible. (b) UV image.

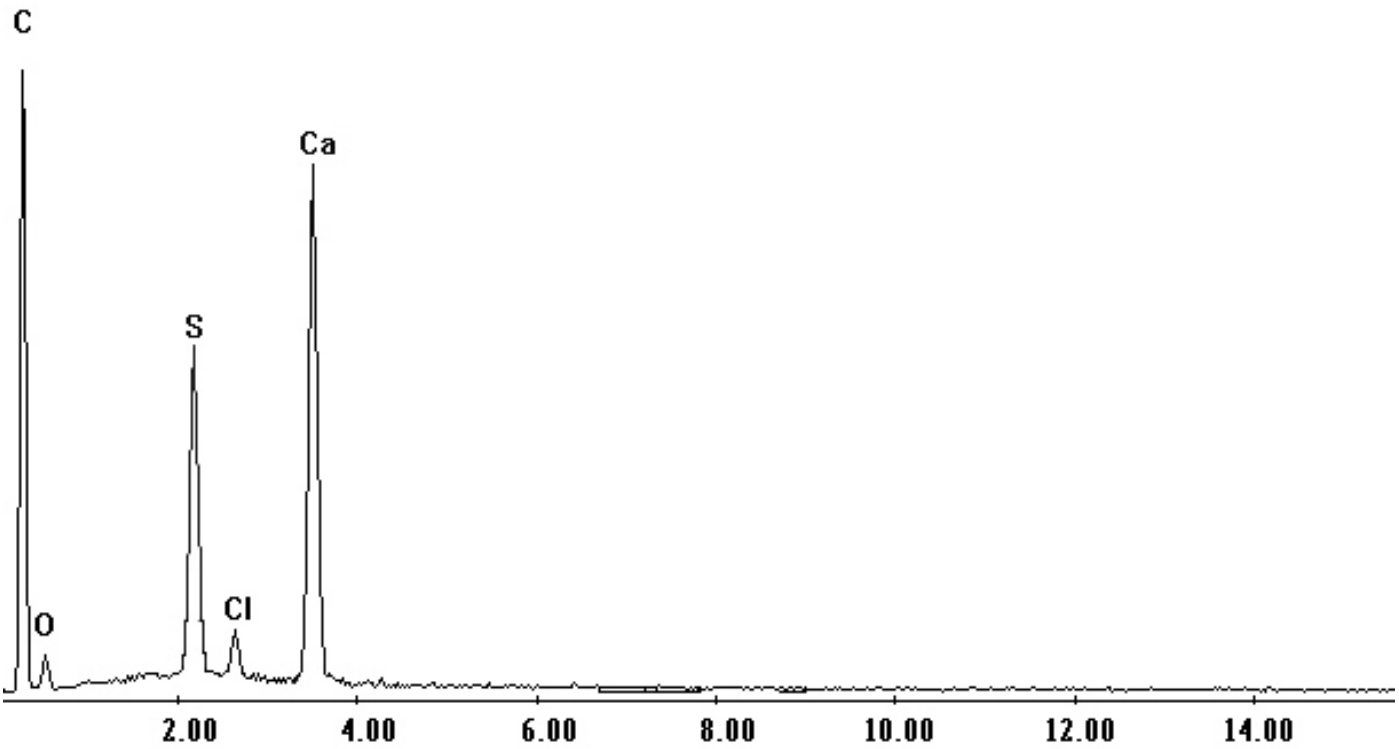

Fig. 6. EDAX results of the black painted layer.

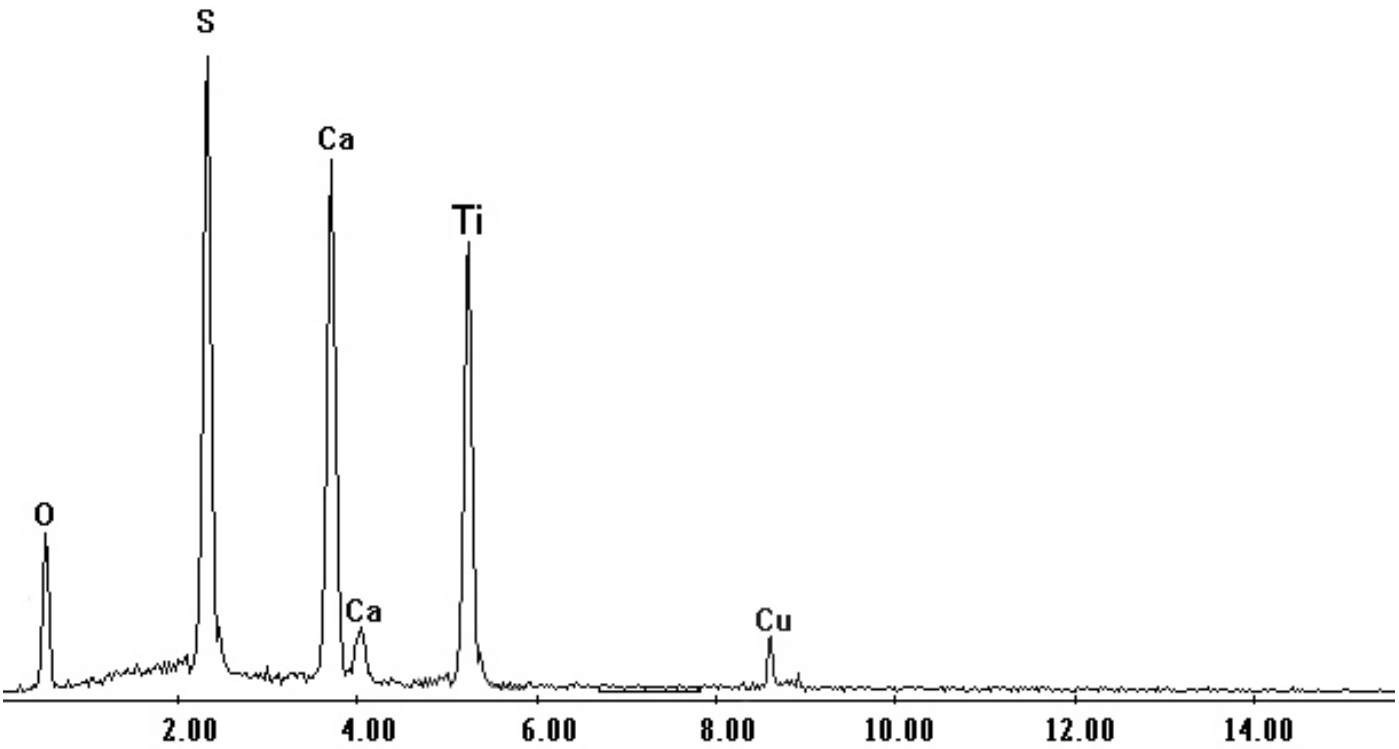

Fig. 7. EDAX results of previous conservation intervention in black areas.

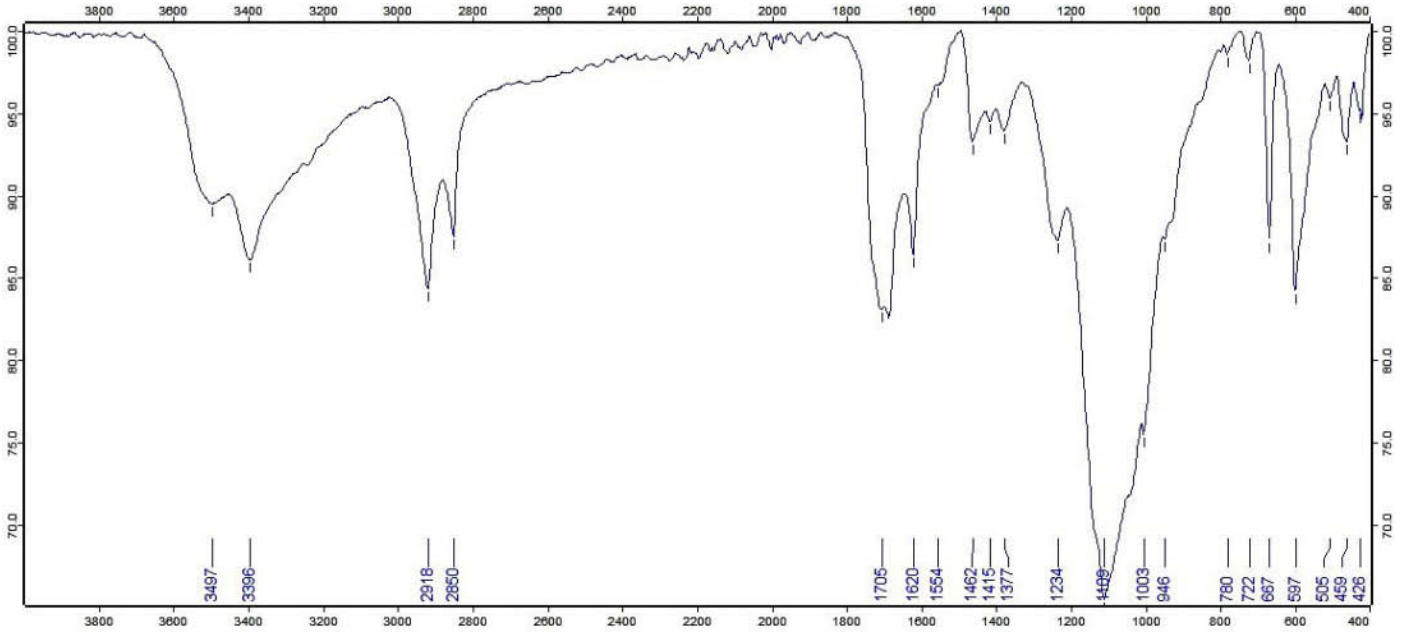

Fig. 8. FTIR results of varnish.

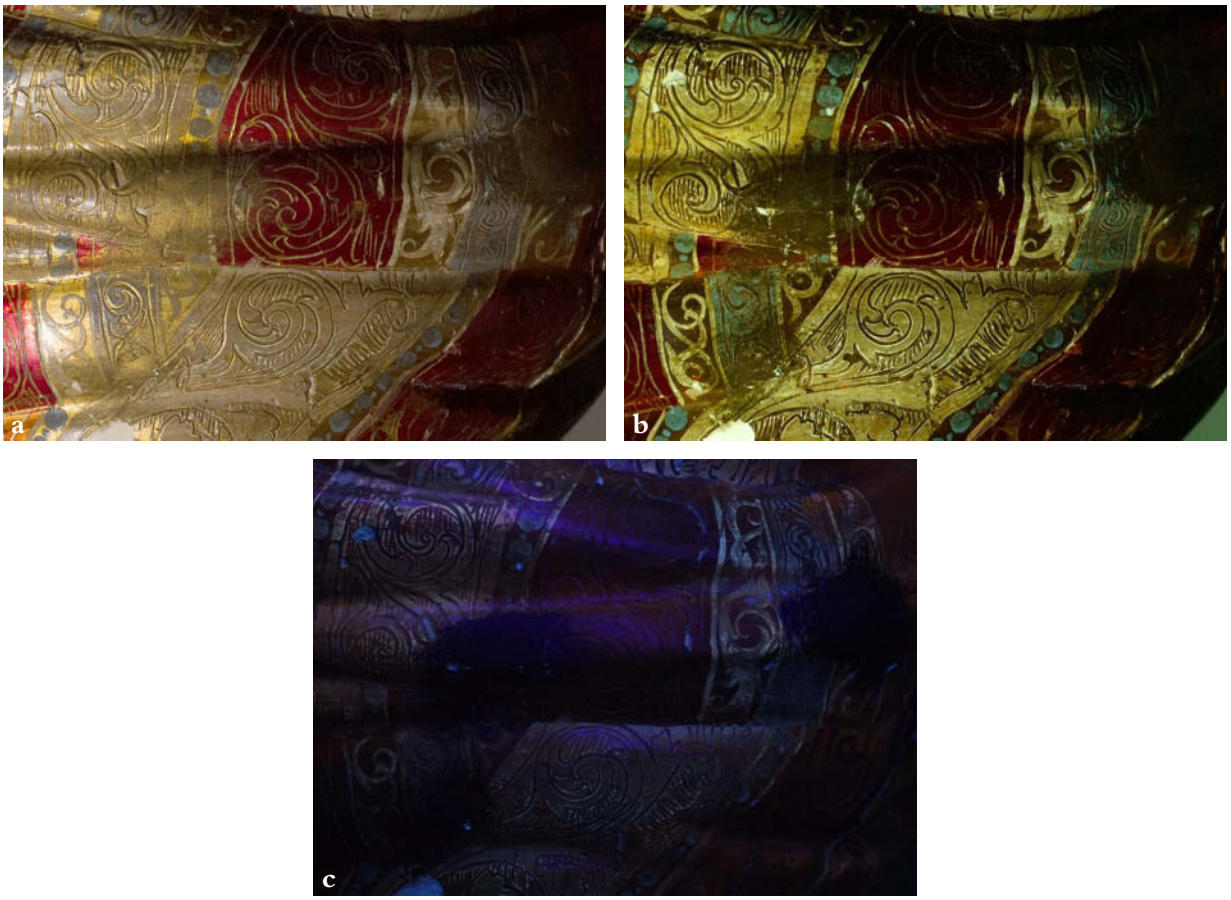

Fig. 9. (a) Visible image. (b) Polarized light image (Hoya FUSION Antistatic CIR-PL Filter and polarization sheets). (c) The UV-induced visible image to differentiate between the original yellow and the retouched.

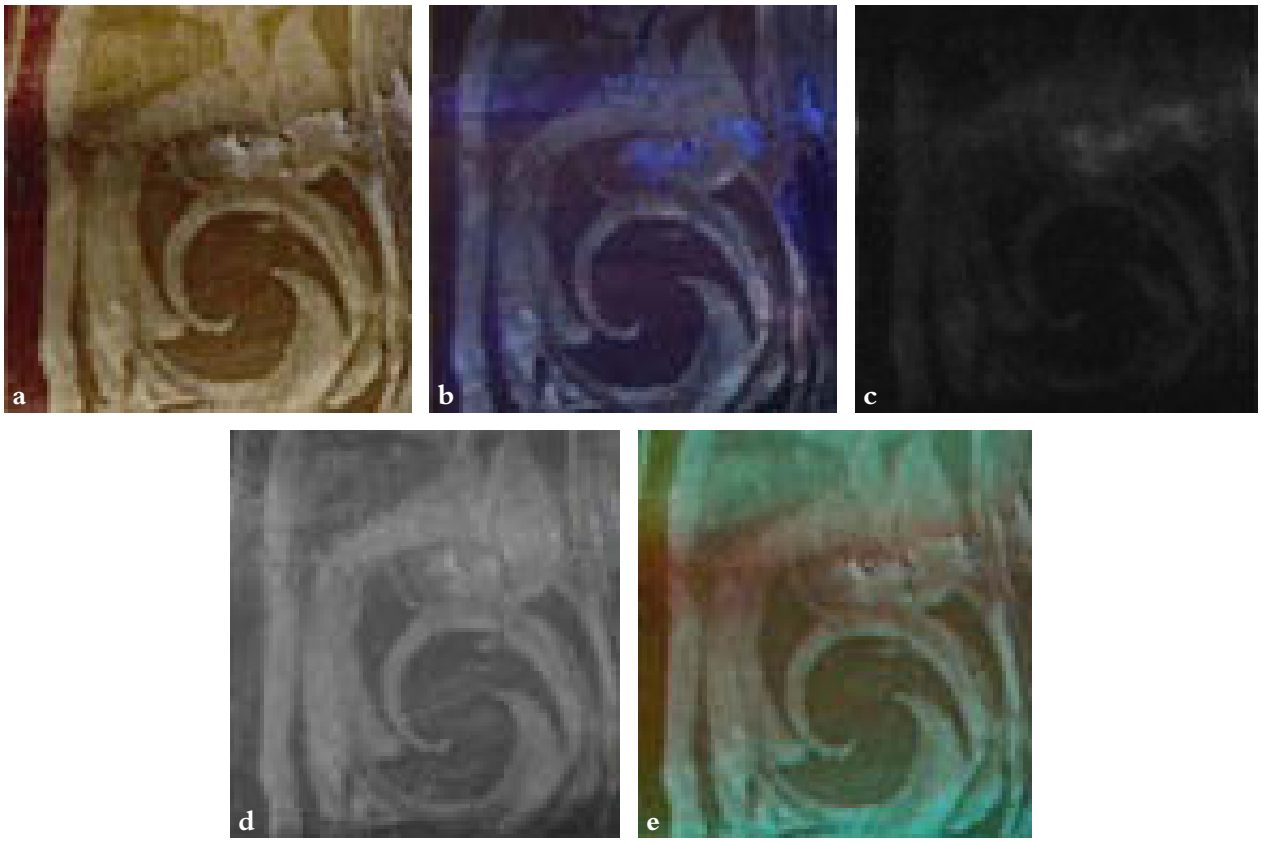

Fig. 10. (a) VIS = retouch. (b) UVL = none/reddish. (c) UVR = dark. (d) IRR = bright. (e) IRFC = white.

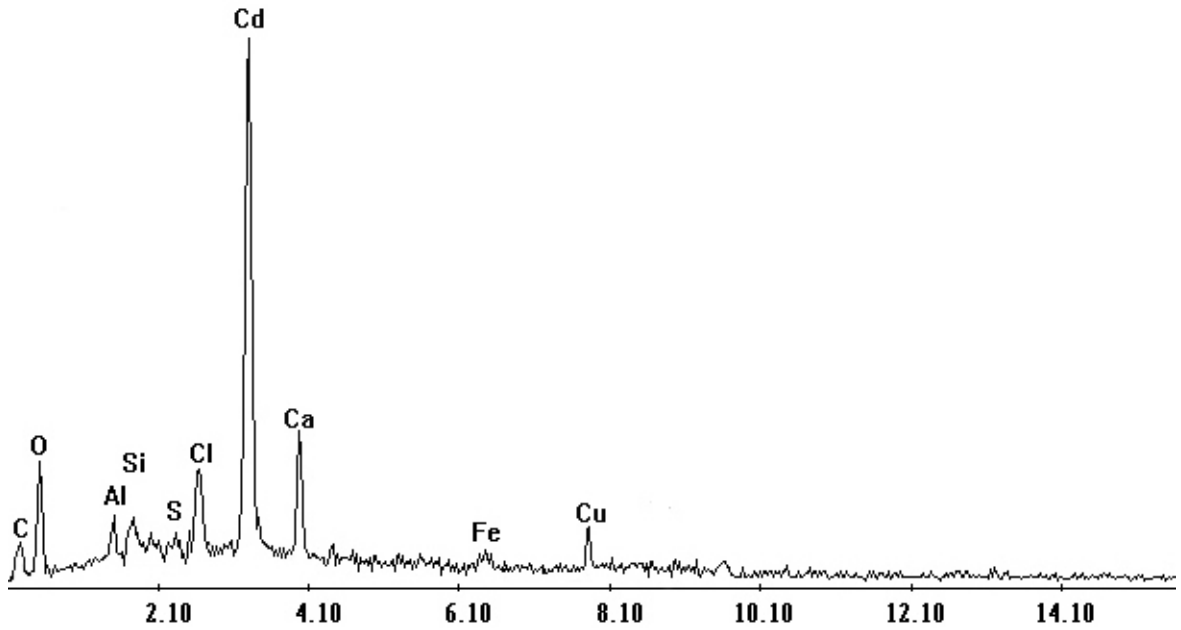

Fig.11. EDAX results of previous conservation intervention in yellowish areas.

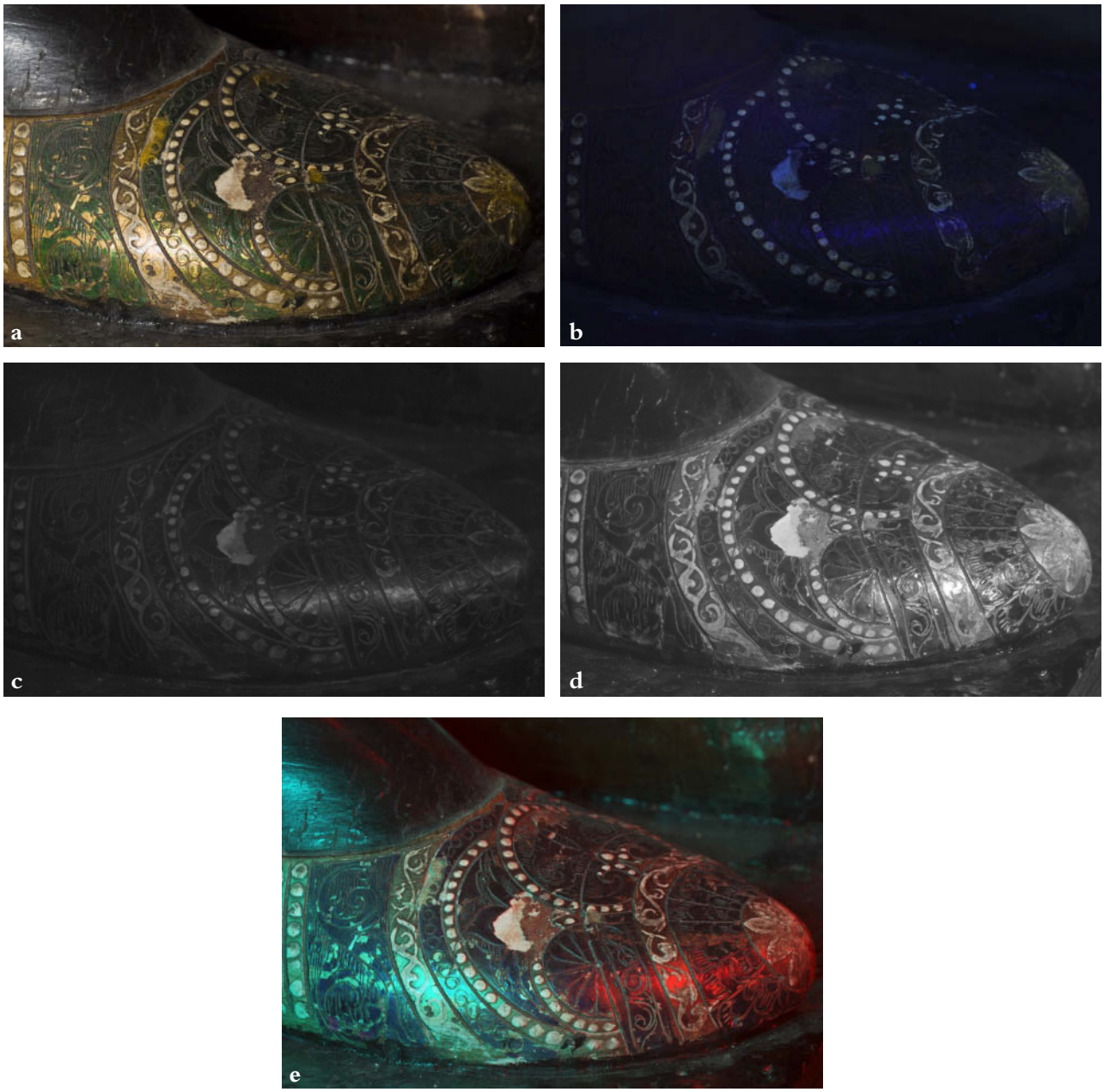

Fig.12. (a) VIS = green. (b) UVL = none. (c) UVR = dark. (d) IRR = dark. (e) IRFC = blue.

in both the UV-reflected image (UVR) and the IR-reflected image (IRR) (figs. 12c, 12d) while showing the blue color in the IR–false color image (IRFC) (fig. 12e). This suggested that the pigment could be malachite (Cosentino 2014). EDAX supported the suggestion of the MSI, which showed high concentrations of copper (Cu), the main component of malachite Cu2CO3(OH)2 (fig. 13).

3.4 IDENTIFICATION OF THE WHITE PIGMENT

EDAX results of the white pigment revealed the presence of lead (pb), the main component of lead white 2PbCO3.Pb(OH)2 (Chaplin, Clark, and Torres 2010), whereas barium, (Ba) the main component of lithopone white (BaSO4.ZnS), was absent (fig. 14). FTIR results proved the presence of lead white: the absorption bands of hydroxyl groups (O–H) appeared at 3403 and 3519 cm^{-1}, whereas the weaker bands were noticed at 1043 and 679cm^{-1}, revealing the carponel group (C–O) (Stanzani et al. 2016) (fig. 15).Processing of the multispectral images (fig.16a) showed no luminescence or slightly yellowish color in the UV-induced visible image (UVL) (fig. 16b). It had a bright shade in the UV-reflected image (UVR) (fig. 16c), a bright shade in the IR-reflected image (IRR) (fig. 16d), and white

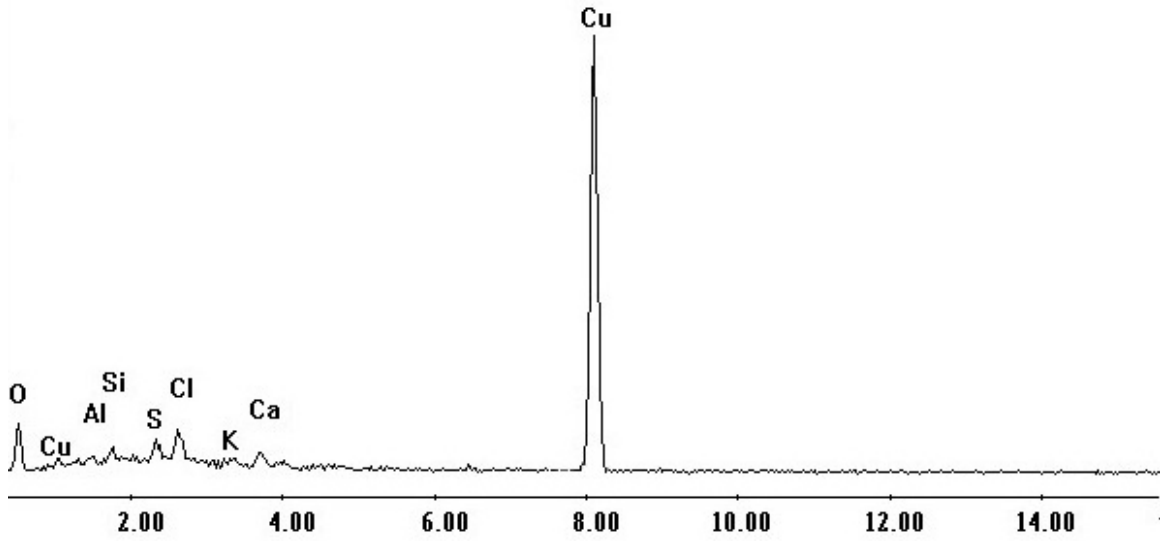

Fig.13. EDAX results of the green painted layer.

color in the IR–false color image (IRFC) (fig. 16e), suggesting that the pigment may be lead white or lithopone (Cosentino 2014).

3.5 IDENTIFICATION OF THE RED PIGMENT

Processing of the multispectral images of the red pigment (fig.16a) showed no luminescence or slightly reddish color in the UV-induced visible image (UVL) (fig. 16b). It had a dark shade in the UV-reflected image (UVR) (fig. 16c) and a bright shade in the IR-reflected image (IRR) (fig. 16d), and showed an orange color in the IR–false color image (IRFC) (fig. 16e), suggesting that the pigment was vermilion, realgar, alizarin, or lead red (Cosentino 2014). EDAX results revealed the presence of mercury (Hg) and sulfur (S), the main components of vermilion (HgS), whereas arsenic (As), the main component of real gar (As4S4), and carbon (C), the main component of alizarin (C14H8O4), were absent. In addition, there was no evidence of the presence of lead (Pb), the main component of lead red (Pb3O4) (fig. 17). All of these results revealed the presence of vermilion as a red painted layer in the reddish areas (Béarat et al. 2005).

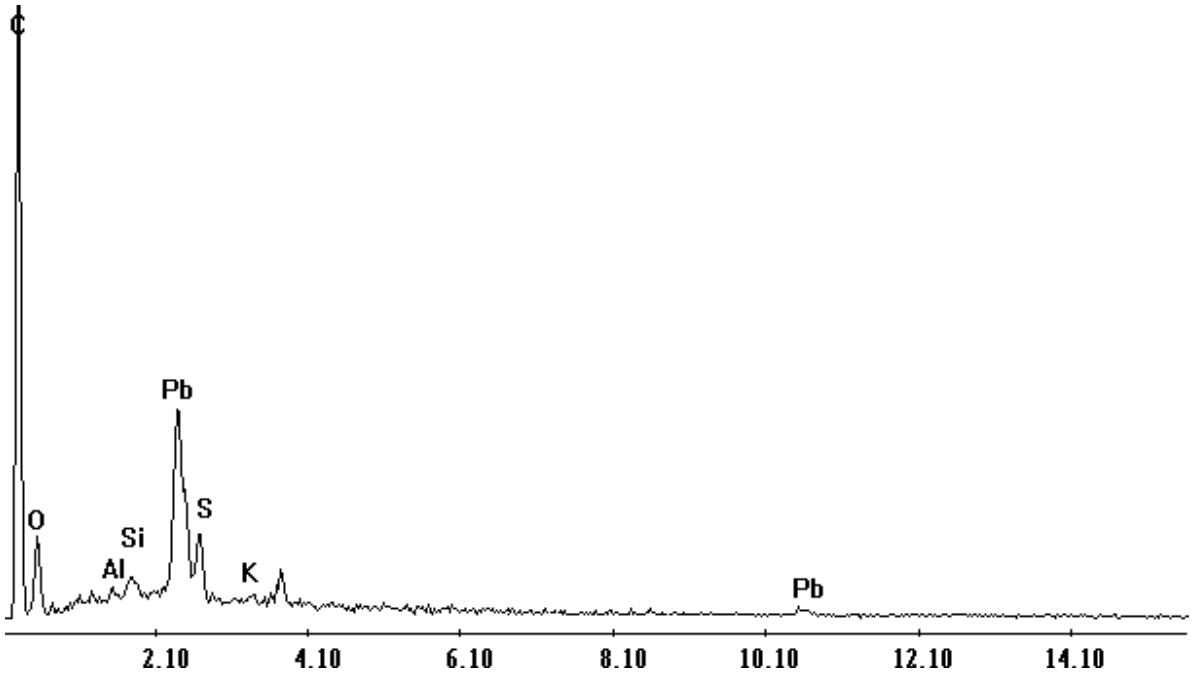

Fig.14. EDAX results of the white painted layer.

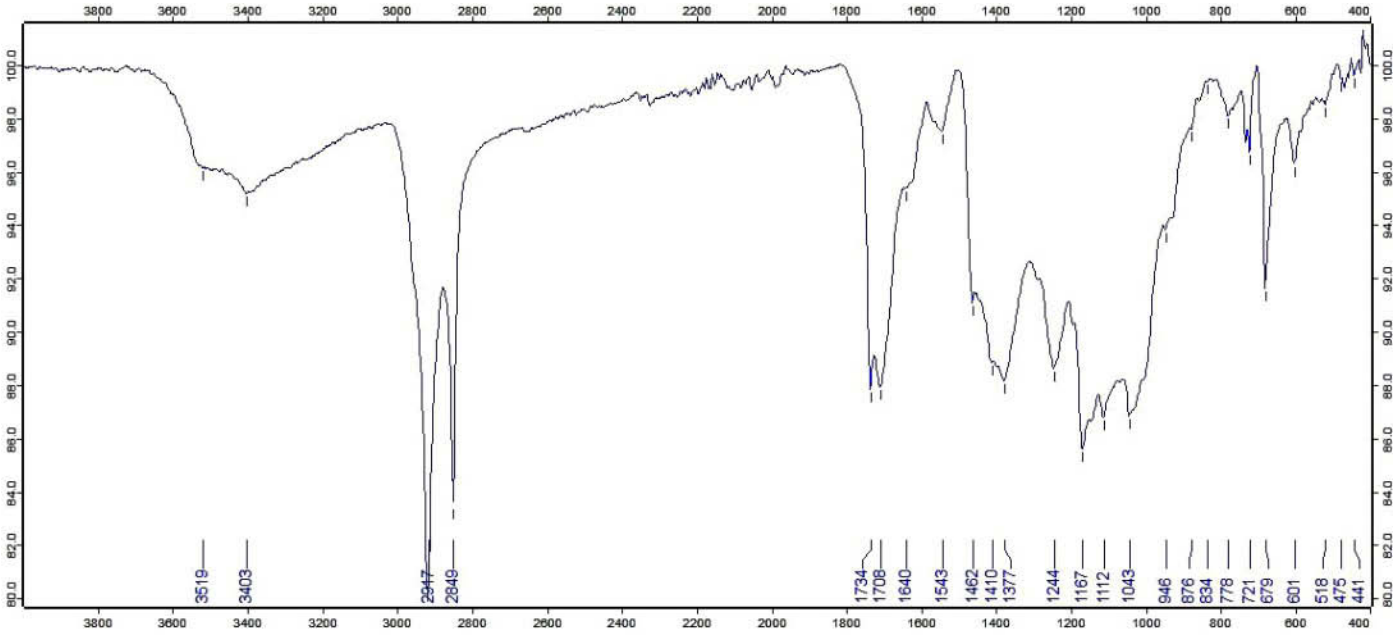

Fig.15. FTIR spectrum of the white painted layer.

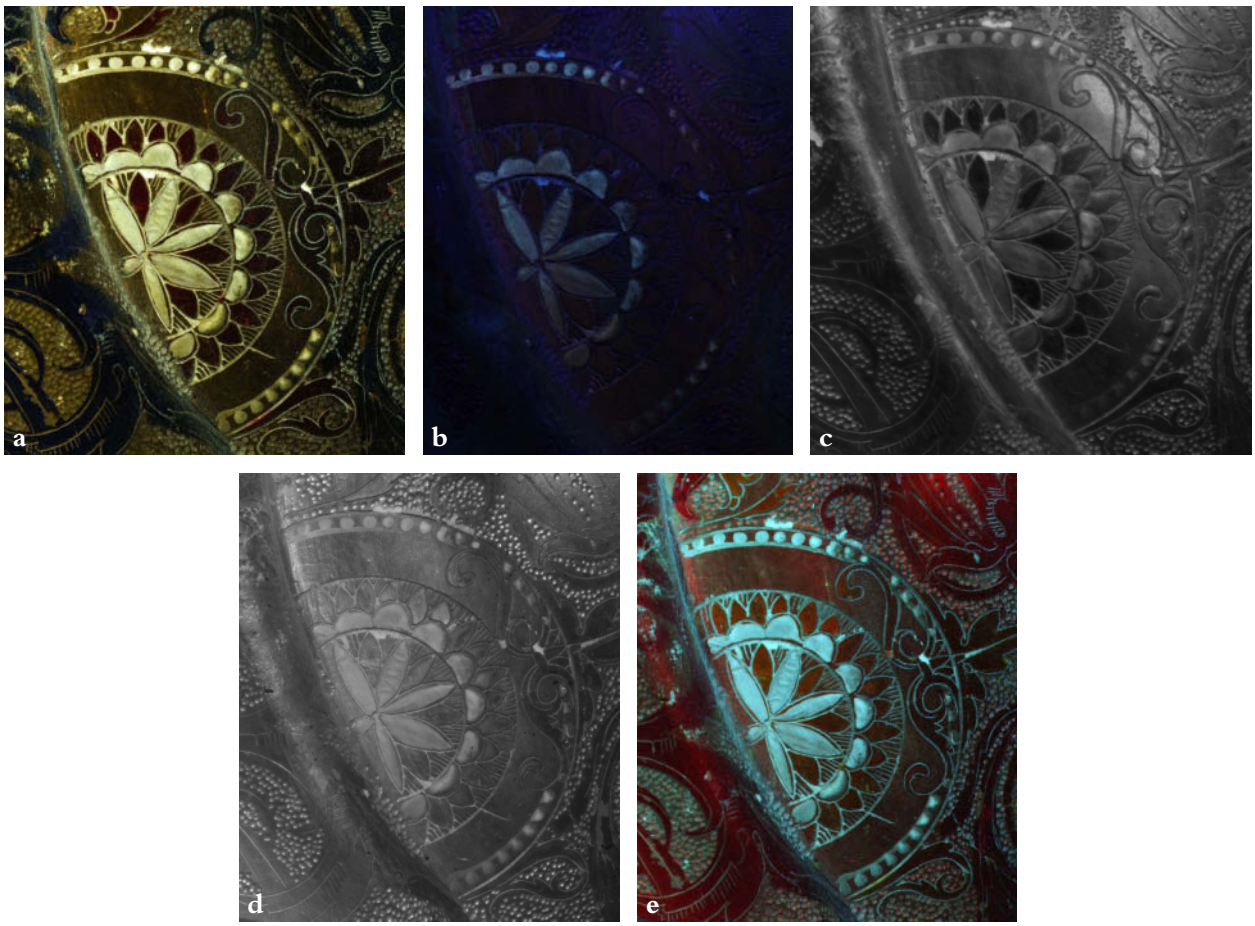

Fig.16. (a) Visible. (b) UV-induced luminance. (c) UV reflected. (d) IR reflected. (e) IR false color.

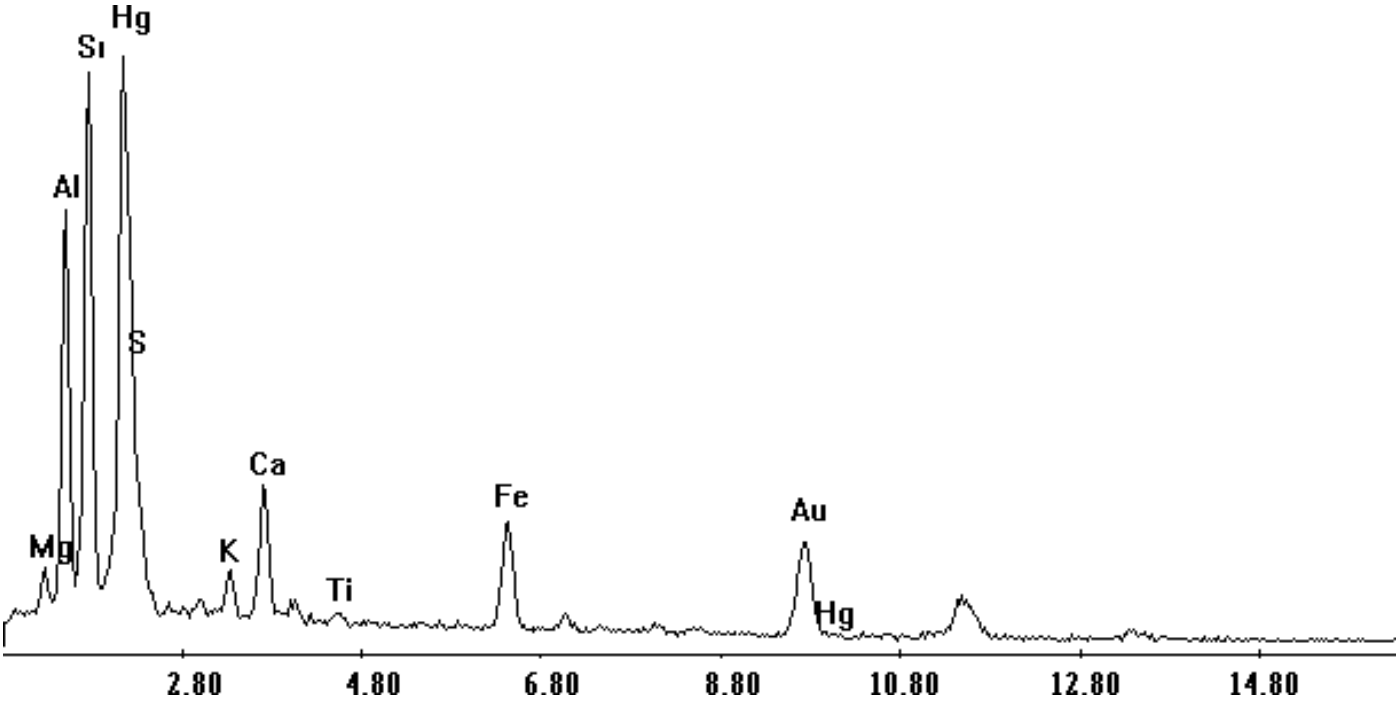

Fig.17. EDAX results of the reddish painted layer.

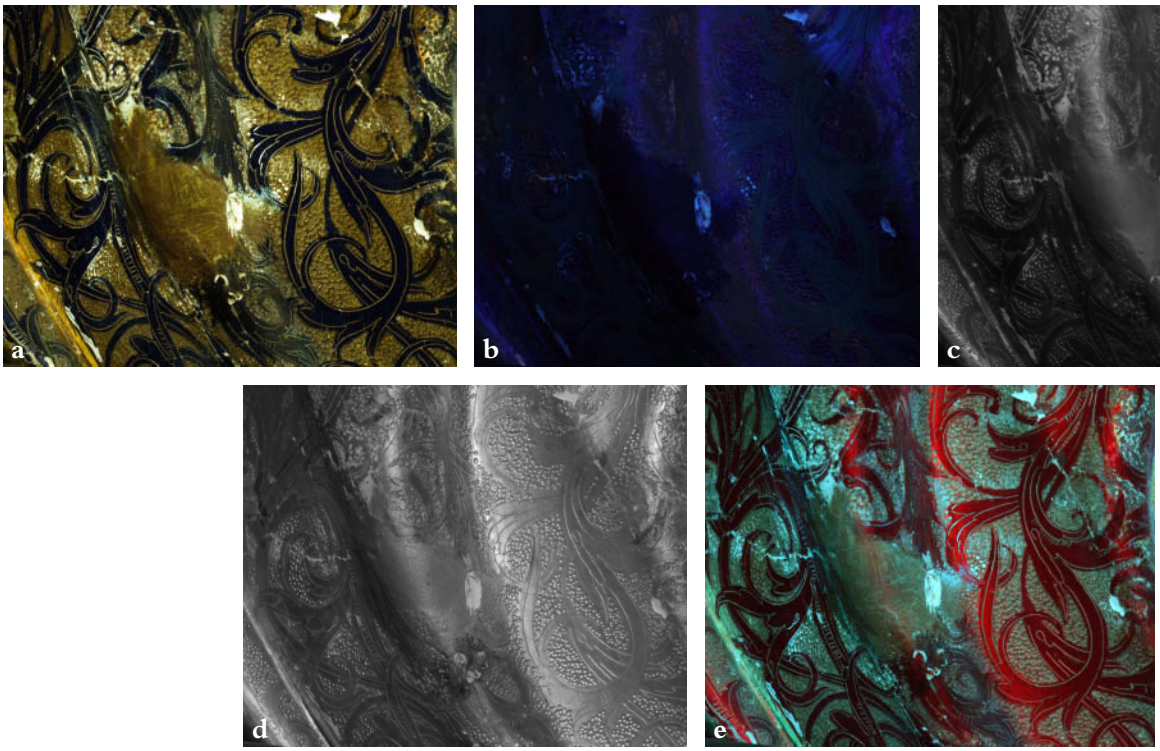

Fig.18. (a) VIS = blue. (b) UVL = light blue. (c) UVR = dark. (d) IRR = bright. (e) IRFC = red.

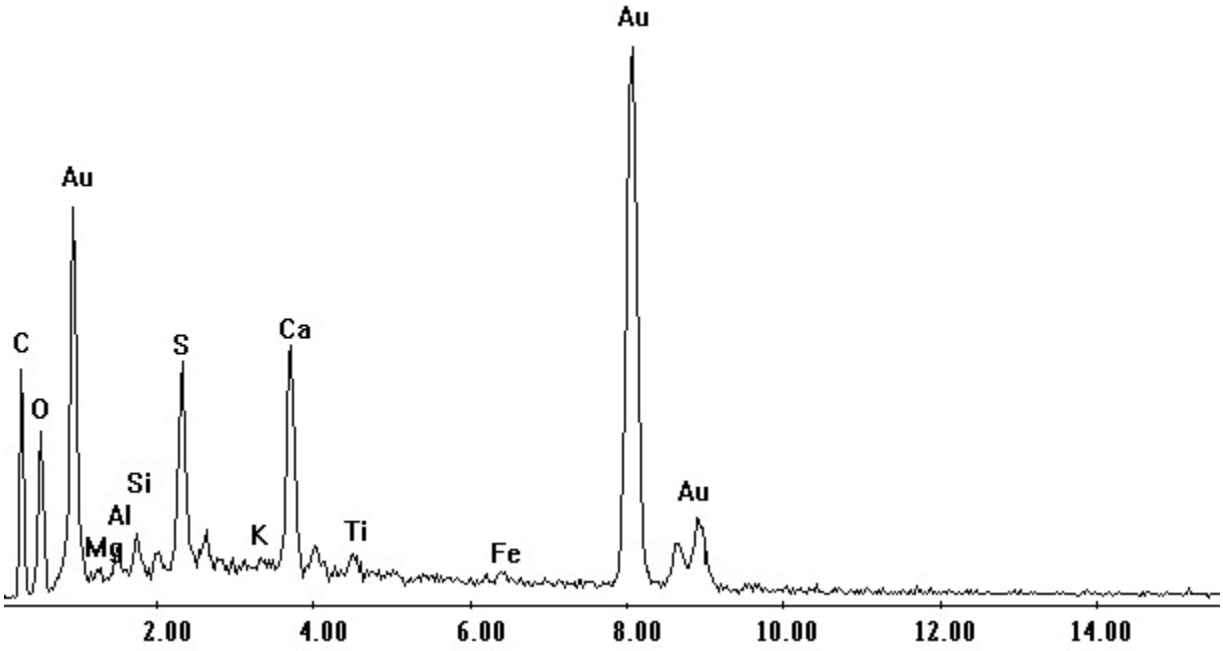

Fig.19. FTIR spectrum of blue painted layer

3.6 IDENTIFICATION OF THE BLUE PIGMENT

Processing of the multispectral images of the blue pigment (fig.18a) showed a light blue-gray color of the pigment in the UV-induced visible image (UVL) (fig. 18b). It had a dark shade in the UV-reflected image (UVR) (fig. 18c) and a bright shade in the IR-reflected image (IRR) (fig. 18d), whereas a red color was present in the IR–false color image (IRFC) (fig. 18e), suggesting that the pigment could be indigo, Maya blue, or phthalo blue. EDAX results revealed the absence of carbon (C) and copper (Cu), the main components of pathalo blue ($C_{32}H_{16}CuN_8$), and there was no evidence of using Maya blue in this period. In addition, the presence of gold (Au) was noted in the gilded layer, whereas calcium (Ca) and sulfur (S) were noticed in the ground layer (fig. 19). FTIR results revealed the presence of indigo as follows. The alkanes (C–H) stretch band appeared at 2850 and 2918 cm^{-1}, whereas the acid group (C=O) stretch band appeared

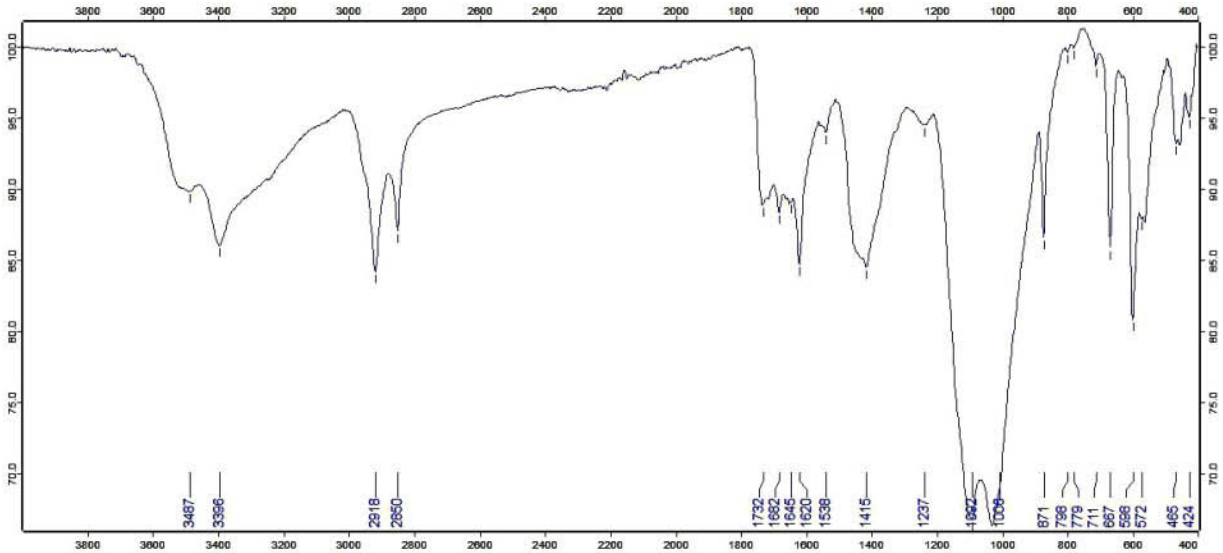

Fig.20. EDAX results of the bluish painted layer.

at 1732 cm^{-1}. In addition, the fingerprint of indigo appeared at 1008 and 1092 cm^{-1}, which revealed the carponel group (C–O) and carbon–nitrogen group (C–N) (Osman, Zidan, and Kamal 2014) (fig. 20).

4. CONCLUSION

This article presented the investigation of a gilded wooden statue from the Ottoman period the combined use of the MSI technique, and SEM-EDAX, as a noninvasive technique to map and identify the painted layers, and previous conservation interventions, as well as complementary techniques such as XRD to identify the ground layer, and FTIR spectroscopy was used in some cases to identify the binding media, and the blue painted layer. In addition, the identification of wood was included in the study. The microscopic observation of wood thin sections allows identifying it as pine wood (*Pinus* spp.). The application of MSI provided useful information about the spatial distribution of the surviving the previous restoration interventions. However, complete characterization of the pigments required the use of other techniques, such as Raman spectroscopy, and XRD. As a result, numerous analytical techniques have found a valuable application in the identification of pigments. However, every single method has particular limitations. For this reason, in most cases it is necessary to employ a combination of several techniques to obtain a complete overview of the composition of a layer of paint.

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RANDY S. WILKINSON

Making Excellent Thin Sections for Wood Identification: A Quick and Easy Method—Part I

ABSTRACT—Making thin sections by hand for microscopic wood identification is a precise exercise with often frustrating results. With microtomes being out of reach for most private conservators, it is difficult to produce good sections that include all desired information. Poor sections result in poor analysis, hence the need for an improved method.

This article explores one such method that has had excellent results. The technique combines a resin (developed for making fish lures and currently also used for forensic analysis), an embedding method for cross sectional stratification analysis, and sectioning with a simplified microtome. The method has three major advantages over conventional systems: it is fast, inexpensive (using simple tools and materials), and reliable, generating thin sections that are large enough for wood identification.

1. INTRODUCTION

This article is the first of two parts and introduces reasons for finding a way to make excellent thin sections for wood identification. The second part delves into the development of a technique.

We would like to start our discussion about making better thin sections for wood identification by beginning with two case studies that illustrate the need for an improved technique to augment the traditional approaches. The first case uses traditional hand techniques and is sufficient to obtain the desired results. In the second case, traditional hand techniques did not lead to a definitive answer; thus, we needed a better method.

Before we look at the case studies, we would like to explain our approach to identifying wood and the steps we take. I developed this three-dimensional model some years ago that begins to frame the discussion. As in all good conservation practice, we begin with the least intrusive approach—that is, a macroscopic inspection, which forms the first side of the pyramid. We observe the grain, color, smell in some cases, and density. On the second side of the pyramid, we use low magnification to observe the grain structure and basic cell structure.

The next side of the model involves connoisseurship. Is the sample we are looking at consistent with craft tradition? Are we sure we are looking at original material? Do the trade routes for the wood in question make sense? Finally, we arrive at the side of detailed microscopic evaluation. On this side, we evaluate detailed cell structure in transmitted light and, in some cases, reflected light. It is here that the process breaks down if we do not have adequate sections.

The first step is a detailed look at the wood in question using a stereoscope, or better yet, a high-magnification digital microscope (fig. 1). I find one with a polarizing filter on it is necessary for evaluating surfaces that are coated. It is in this step that one may be able to learn a good deal about the wood structure, making sectioning unnecessary at times. It can also provide information about cell structure on certain planes that make sectioning that plane unnecessary as well.

The next step in the process is selective sampling of the object, where one only removes a thin section from a certain plane. For instance, let us say that softwood is suspected, perhaps the white pine group. Visual inspection confirmed resin canals and a very gradual transition of early wood to latewood can be seen. If a radial surface is exposed, a simple, barely detectable hand sample can be taken and evaluated under high magnification to confirm smooth wall ray tracheids and large window cross-field pitting. In this case, the sampling is minimal, effective, and the answer is obtained.

Following the preceding steps, the next step in the process would involve removing a sample from the wood in question for analysis in all three principal planes. It is here that hand sectioning can be highly effective but also disappointing. In general, the less dense woods are easier to section by hand than the denser woods, like rosewood. If the desired results are not sufficient, a clearer, cleaner, and planer sample is necessary. The final step is to embed the sample. It is here that a microtome is not always available, and therefore an alternative was developed.

Now that the thought process and the steps involved have been presented, let us turn to our two cases. In the first case, a hand section was sufficient, even though it could be improved by having larger and more even sections. In the second case, hand sections were not sufficient.

2. SPOONER CHEST OF DRAWERS

Last year, I was asked by Brock Jobe to evaluate the inlay on this wonderful chest of drawers, attributed to Alden Spooner (1784–1877), as seen in figure 2. The question was straightforward. Is the banding on this chest ash or sumac? The grain structure is consistent with both choices, namely ring porous, no large rays visible, and so forth. The color of ash and sumac are close, even in their unstained or varnished appearance, although sumac can

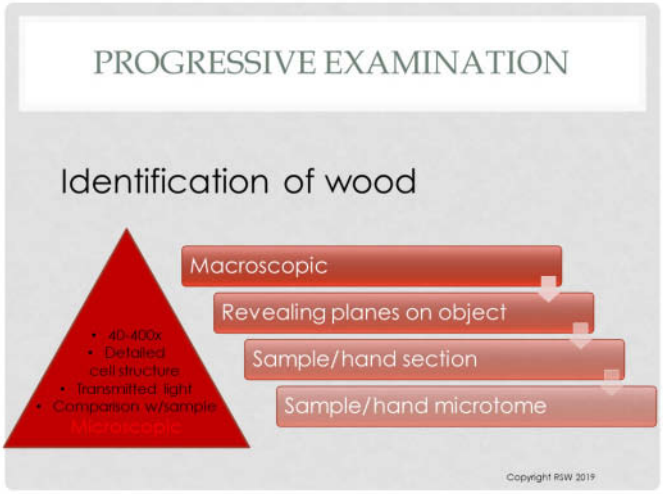

Fig. 1. Progressive process for wood identification.

Fig. 3. Ash versus sumac.

have a bit of a light brown to gray/green look, in contrast to the creamy white color often associated with ash, but they do look similar.

Both are ring porous with approximately the same early wood vessel diameter and number of rows, but it is in the latewood that we draw some distinction. The latewood vessels in ash are relatively few compared with that of sumac, and if we look closely, there is a greater concentration of latewood vessels in the later part of the latewood in sumac, which is not present in ash (fig. 3). Therefore, the goal was to learn (if we could obtain a small sample to compare the cell structure to our known samples) if we would be able to answer the question.

Luckily for me, there was a small piece of the banding that was delaminated on the proper right side of the chest. This occurred at the base level. This was the best-case scenario possible; the banding was easily removed with no damage and could be evaluated, then reattached to the object (fig. 4).

Making a good hand section from this material was almost impossible without destroying the small fragment. Viewed in reflected light, the transverse section more closely matched that of sumac, but I did not have a clear view or a full growth increment. Thus, I removed a small thin section from the side of the banding, and even though the information was fuzzy and slightly out of plane, it did reveal the one detail, specifically the presence of helical thickening in the vessels that allowed for the positive identification of sumac (fig. 5). One last thought was to confirm my assumption of sumac based on the presence of helical thickening by placing the sample under UV light. If it were sumac, it should fluoresce a bright yellow, whereas ash does not fluoresce at all. I placed the banding alongside my known sample of sumac, and both fluoresced a bright yellow. So we had our answer; the banding on the chest was sumac. The point of sharing this case is that sometimes one gets lucky. With a narrow question, a combination of a great sample location, and using a variety of

Fig. 2. Chest of drawers 5.38.11 by Alden Spooner (1785–1877). Courtesy of Old Sturbridge Village, photograph by Gavin Ashworth.

Fig. 4. Sample taken from the base inlay on the proper right side of the chest.

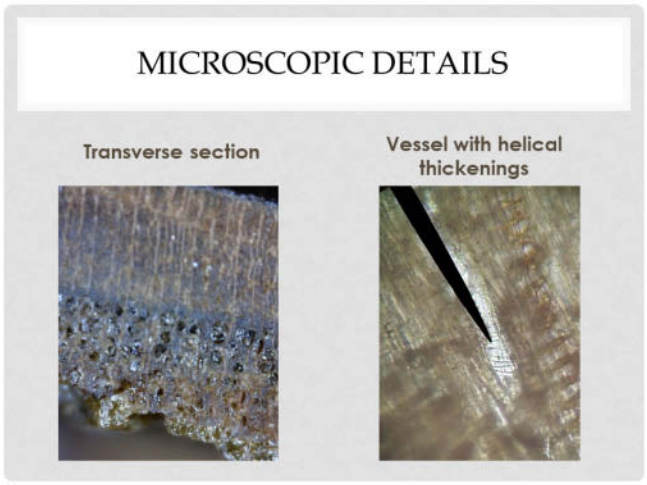

Fig. 5. Helical thickenings present in the vessel element.

techniques, even a poor thin section can be useful and sufficient.

3. DESHON BUREAU TABLE

Now let us turn our attention to a case in which a simple hand thin section was not sufficient to make an accurate wood identification. In fall 2017, the bureau table as seen in figure 6 was on exhibition at the Yale University Art Gallery in New Haven, Connecticut. It was part of the exhibition Art & Industry in Early America; Rhode Island Furniture, 1650–1830. I had the unique opportunity to attend a 2-day forum that included scholars, curators, collectors, and professionals for an in-depth look at the treasures in this exhibition. It was at this event that I first looked at the Deshon table with a critical eye. My initial impression was that the color of the wood was unlike that of mahogany—very pale in comparison to the other objects in the exhibition. Reading the description, the wood was described as "blond mahogany." After taking a closer look, I concluded that one of two things was true. Either I really did not know what mahogany looks like from the cell structure or this was not mahogany as it has been assumed to be and described since the chest's fabrication in 1764. The second thing I noticed was that the pores size did not match the relatively small and diffuse pattern of mahogany. The pores were very large and few per millimeter. In addition, the inside of a drawer had relatively wide bands of pigmentation lines. It was at this point I suggested to Curator Patricia Kane that we seek permission to sample the table for wood identification after the exhibition closes.

After receiving permission to sample the chest, a more detailed inspection was in order. My first observation was that we had a very clear and clean view of the exposed pin on the drawer side. If we now compared the Deshon sample with that of a known sample of Swietenia, the sample did not match the cell structure of mahogany. The macroscopic features that are inconsistent with mahogany are the presence of banded parenchyma; dark pigmentation lines; barely visible ray; and, by comparison, very low density. These can clearly be seen in figure 7.

It was at this point that the removal of a wood sample was necessary for traditional wood identification. Therefore, I removed the lock from one of the drawers and removed the sample along an existing cut in the wood behind the lock plate.

One of the first observations after removing a tangential section by hand from the sample was the confirmation that the cell structure of the rays was inconsistent with mahogany. However, the sample did confirm the presence of uniseriate rays that were unstoried; a feature not found in *Swietenia* spp. Unfortunately, my hand sample was not in plane across the entire field of view under the microscope due to poor sectioning of the sample. Yet, fortunately, my sections got somewhat better for the other principal planes, and I was able to discern and confirm many other features. However, I did not have enough information to make a positive identification of the wood; I was missing something,

Fig. 6. Bureau table 1765, private collection. Photograph RIF685 courtesy of Yale University Art Gallery.

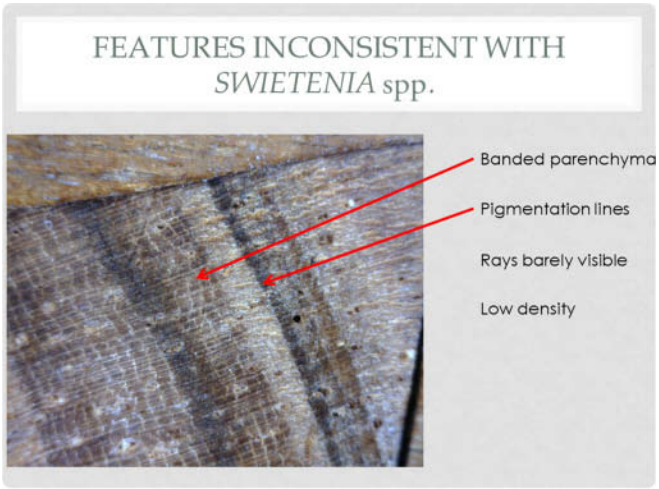

Fig. 7. Features inconsistent with *Swietenia* spp.

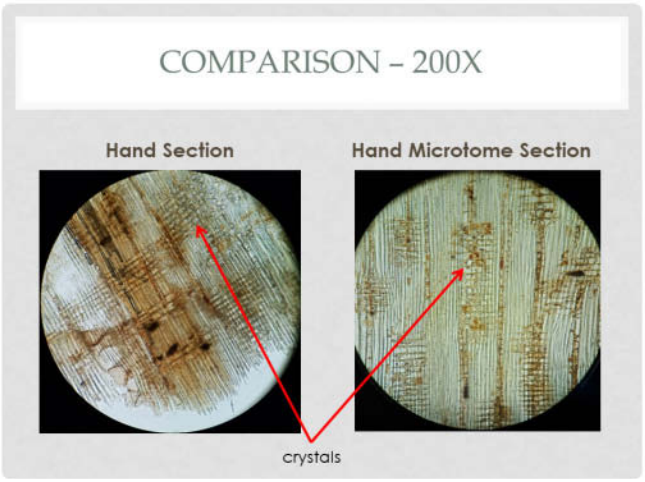

Fig. 8. Comparison of a hand section versus a handheld microtome section—radial 200x.

and this object was too important to make a mistake. It was at this point that I sought the help of Dr. Regis Miller.

I sent the sample to Dr. Miller with a list of what I thought were positively identified cell features for the sample. Note here that even with poor to relatively good hand sections, I was able to furnish Dr. Miller with a lot of information.

The one key feature I missed was the presence of crystals in both the rays and the axial parenchyma. On the left hand side of figure 8, we see an image of my hand section taken again after getting the sample back from Dr. Miller, and it can be observed that the section is not terrible but uneven and the crystals can be easily missed. What is seen on the right hand side of figure 8 is an image of the radial section, made with a hand microtome. This image provided a clear planer view, and the crystals are easily observed.

The "blond mahogany" was as I suspected—not mahogany at all but rather manchineel (*Hippomane mancinella L.*), and the reason I was not able to arrive at this conclusion was due to poor hand sections. Quite honestly, at the time of sampling, I did not have enough experience observing crystals in both ray and axial parenchyma cells.

4. CONCLUSION

The preceding two case studies show that one can arrive at satisfying conclusions for wood identification by a variety of means, sometimes even without actual sectioning. However, when there is a need to section, one would like to have the information as clear as possible. The last case study showed that better sections would likely have led to a faster identification, prompting the need for an improved sectioning method.

Please refer to Rian Deurenberg-Wilkinson's article on the subsequent development of a technique to make better thin sections for wood identification.

AUTHOR BIOGRAPHY

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RIAN M. H. DEURENBERG-WILKINSON

Making Excellent Thin Sections for Wood Identification: A Quick and Easy Method—Part II

ABSTRACT—Making thin sections by hand for microscopic wood identification is a precise exercise with often frustrating results. With microtomes being out of reach for most private conservators, it is difficult to produce good sections that include all desired information. Poor sections result in poor analysis, hence the need for an improved method.

This article explores one such method that has had excellent results. The technique combines a resin (developed for making fish lures and currently also used for forensic analysis), an embedding method for cross sectional stratification analysis, and sectioning with a simplified microtome. The method has three major advantages over conventional systems: it is fast, inexpensive (using simple tools and materials), and reliable, generating thin sections that are large enough for wood identification.

1. INTRODUCTION

This article is the second of two parts and delves into the development of a technique to make excellent thin sections for wood identification. The first part, by Randy S. Wilkinson, introduced reasons for needing a better technique.

The ideal sample for wood identification is thin, even, and includes all desired information, such as a complete annual ring. It should also be exactly transverse, radial, or tangential. Figure 1 shows an example of a section made with the hand microtome method that this article describes and that meets all of the preceding requirements.

Unless one has access to (and experience with) a microtome, one would section by hand. Hand sectioning has two major benefits: it is inexpensive and fast. However, depending on one's skill level, it is hard to make sections that are thin and even. When making a thin section, one often needs multiple slices to make up the complete picture of an entire annual ring, vessel perforation plates, parenchyma patterns, and so forth. There are multiple techniques for holding the material and the blade, but they all take quite a bit of practice to make good sections.

With a rotary microtome, one can make larger sections that are very even. However, microtomes are an expensive purchase and require sample embedding (and rinsing), experience with operating it, and specialized sharpening of the blades. A hand microtome may aid in guiding and steadying the blade, but most hand microtomes that are sold commercially have a large opening and still need a system for holding a wood specimen down and steady while making a cut (fig. 2). A wide variety of blades can be used with a hand microtome, from large old-fashioned razor blades to single- or double-edge razor blades, or a chisel.

Considering that the premise of a low-tech way to guide the blade and obtain larger sections was interesting, a better version of a hand microtome was developed. There were two areas that could use improvement for this purpose. First, the specimen embedding method needed to be fast and clean, and preferably not require rinsing of the sample. Second, the opening in the specimen holder needed to be much smaller. The right type of cutting blades was a third consideration.

2. EMBEDDING RESIN

Embedding a sample before sectioning ensures the proper shape for clamping and mounting in a microtome and can also help reduce fraying or breaking of more fragile material.

It turned out that the Tuffleye embedding resin we currently use for preparing samples for cross sectional stratification analysis is soft enough to be cut with a razor blade after curing. The embedding method with Tuffleye was developed by James Martin, then at Orion Analytical LLC but currently at Sotheby's as director of scientific research. The Tuffleye resin is a blue light (not UV)-cured acrylic resin (aliphatic urethane methacrylate blend) that sets in 10 to 30 seconds. The resin was developed for making fish lures but has had attention in the forensic field because of its quick cure time and clear color (Groves and Palenik 2016). The specimen is mounted on the cap of a bullet-shaped plastic embedding capsule, after which the capsule is filled up completely and cured with a blue light (fig. 3). The shape of the capsule and the insertion of the syringe tip in a narrow hole at the top aids in spreading the resin. Because the small hole is closed off by the syringe, the resin is forced all around the specimen, decreasing air bubbles. Curing the resin-filled capsules on a mirror can help distribution of the blue light. Once the resin is cured, the embedded sample can usually be removed by pushing it out with the aid of a skewer, saving the capsule for future use. Alternatively, the capsule can be cut open

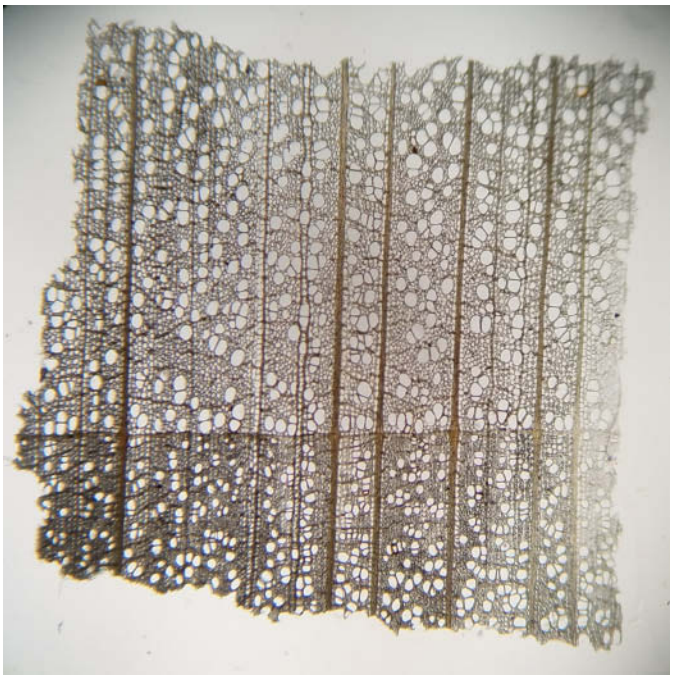

Fig. 1. Thin section made with hand microtome, basswood, transverse, 40x.

with a razor blade. The residual stickiness on the surface of the embedded samples can be wiped off with ethanol. Even wood that was damp after boiling until it sank could be embedded without a problem.

For smaller specimens, usually radial and tangential orientation, multiple sections of the same orientation can be glued together with superglue to form a stacked specimen. This will provide more volume and prevent the specimen from getting dislodged from the resin during slicing. If multiple blocks are not available, one can superglue any other orientation or species, such as a section of bamboo skewer, to the specimen to provide sufficient length and volume for secure embedding.

As with a rotary microtome, it is very important to have the orientation exactly right. Once the wood sample is embedded, the orientation cannot be changed. It is therefore prudent to be extremely precise during the embedding process to have one of the three orientations as perfect as possible.

3. HAND MICROTOME

Having found a satisfactory, quick way to embed the samples, a hand microtome was developed that would fit the cylindrical-shaped samples. The first prototype of a homemade hand microtome used a regular 5/16 in. coupling nut for connecting threaded rods. One side was drilled out to remove the threads. The cylindrical embedded specimen fit in it well, but nothing prevented it from being lifted up when slicing. In addition, the thread of the screw to turn up the specimen was too coarse, making it hard to accurately and consistently produce an even slice. A coupling nut with a finer machine thread was not a significant improvement. The bolts also had too much slack to stay stationary during slicing (fig. 4).

Fig. 2. Example of a commercially available hand microtome.

Fig. 3. Embedding wood specimens in plastic capsules with the Tuffleye Core embedding resin.

Fig. 4. Different prototypes for a hand microtome, made out of coupling nuts.

Next came a smooth shaft coupling, used for connections in machinery (fig. 5). A small micrometer was mounted on one end, whereas the other side held the specimen. Turning the knob on the micrometer advanced the specimen in a controllable and measurable way. A pair of set screws on either end of the coupling provided stability of the micrometer as well as the sample. The coupling was made of stainless steel—a more durable cutting surface than aluminum. The small cutting table avoided unnecessary dulling of the razor blades. This new model proved to be much more controllable as far as sample advancement and security within the holder. It was fairly easy to produce multiple large slices that were extremely thin and even.

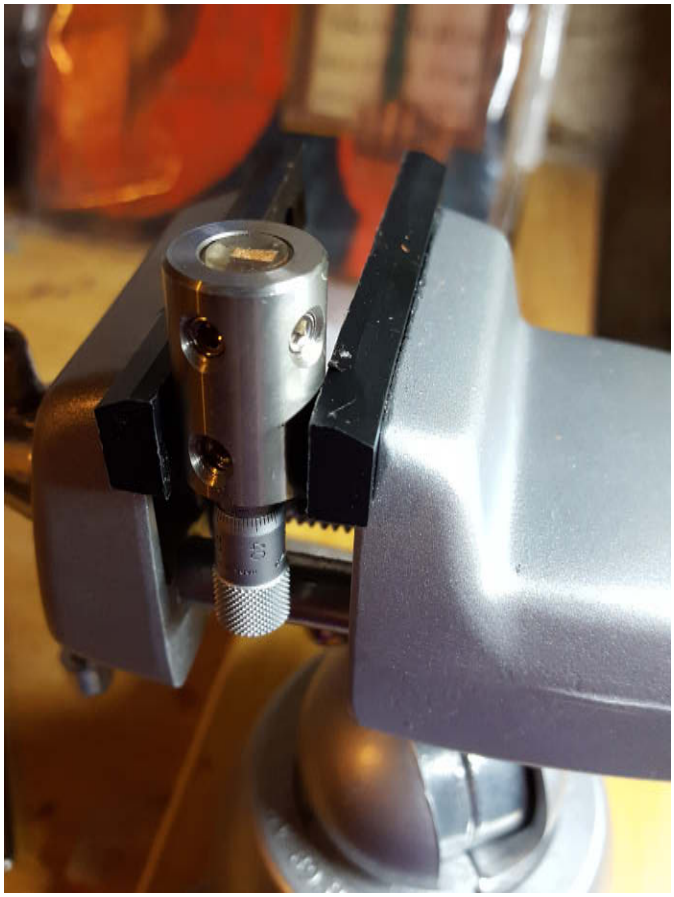

Fig. 5. Final version of the hand microtome, made with a small micrometer and a smooth shaft coupling.

4. BLADES

Another consideration while developing the method were the blades to cut the thin sections. Both the regular and more expensive razor blades from a hardware store did not last. They dulled very quickly and sometimes did not even make a clean cut straight out of the box. The results were often unsatisfactory with ripped or distorted sections. The Forest Products Laboratory in Madison, Wisconsin, uses GEM blades, which were a great improvement as far as being—and staying—sharp. GEM and PAL are well-known brands that produce several types of blades for different scientific applications. The GEM PTFE (polytetrafluoroethylene [or Teflon]-coated) single-edge blades that slide through the resin easily became a favorite. For transverse sections, especially harder woods, the heavy-duty carbon steel blades sometimes produced a more even thickness and a cleaner cut. Although it was easier to hold the blade in a razor blade holder, it did not allow for a sufficiently low cutting angle.

A dedicated, well sharpened chisel still needed occasional sharpening and in general was not an improvement over the hardware store razor blades. A regular utility knife blade was nice and rigid but not sharp enough, whereas flexible double-edge razor blades were so flexible that the sections would be "scooped" out of the resin, producing slices that were markedly thicker in the middle.

5. SLICING

The best practice for slicing was to apply a drop of water with a pipet and use a sideways slicing motion to cut a section. A smooth sweeping motion rather than a sawing action yielded the best results. Once a clean-cut surface was established, the micrometer would advance 50, 75, or 100 µm, depending on the hardness of the wood and the grain direction. Before slicing, it was necessary to tighten the set screws on the side of the specimen to keep it from being lifted up during slicing. One can divide the blade in three sections by writing numbers 1 through 3 on them with a permanent marker and only use each section for one or two slices. This way, one does not use an already dulled portion of the blade.

The literature suggests 15 to 25 µm for a good wood section (Schoch et al. 2004). However, even the 50-µm advancement would usually not allow for a proper cut. Despite the larger advancement of the micrometer, the sections had the correct thickness for wood identification.

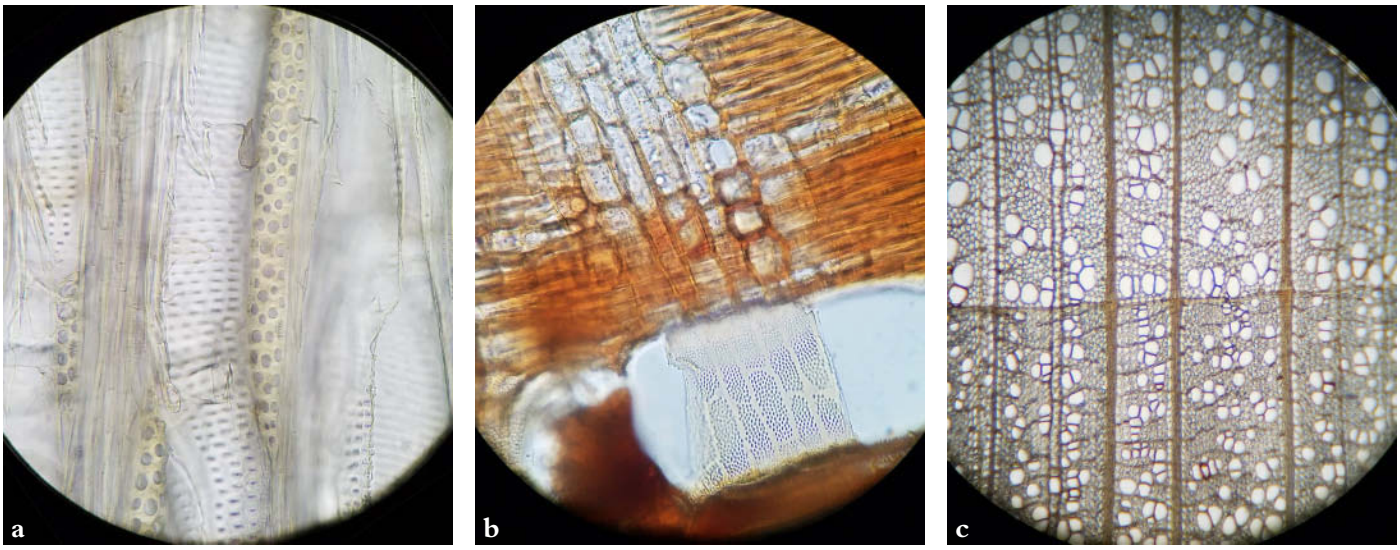

Fig. 6. (a) Poplar, tangential, 40x. (b) Mahogany look-alike, radial, 40x (note the particularly nice example of ray-vessel pitting). (c) Basswood, transverse, 10x.

6. ALTERNATIVES

When sample size and location are not an objection, an 8-mm plug cutter can be used to make a cylindrical specimen in tangential, radial, or transverse orientation. This saves the time to embed the samples, but one can still use the hand microtome to guide the razor blade and make sections. There is more wood loss, however, and the correct orientation would need to be checked before drilling. The cylinder can usually be removed from the substrate by wiggling it until it breaks at the base rather than cross cutting it with a saw.

Paraffin is the most common embedding medium for samples in preparation for slicing on a microtome. However, paraffin is messier, leaving wax slivers all around the cutting area in addition to a waxy residue in the sections. The residue needs to be removed by several solvent rinses, which adds extra steps and time to the process. If not properly removed, the residue presents itself as gray matter in the cell cavities. Another disadvantage of the paraffin is its softness, allowing a more flexible blade to dig too deep and produce sections that are too thick, especially in the middle. A heavy-duty, thicker blade yields better results.

7. PREPARING THIN SECTIONS FOR VIEWING

Once several good sections were cut, they were prepared for mounting on a microscope slide. First, the sections were removed from the surrounding embedding resin with a needle or scalpel blade, leaving only the specimen on the slide. They were all rotated to have the same orientation on the slide, making it easier to scroll through them during viewing.

A cover slip was laid over them, and a small drop of ethanol/glycerin (1:1 ratio) was wicked under the slip. Air bubbles were subsequently boiled off on a hot plate. Making a simple U-shaped aluminum foil tray for the slides aided in easy removal from the hot plate without burning one's fingers.

Alternatively, one can mount the sections more permanently in Cytoseal 60 or 280.

Figure 6 shows a few examples of sections made with the hand microtome: a tangential and transverse section of poplar on either side of a radial section of a mahogany-like wood. The ray-vessel pitting in the middle section is a particularly nice example of the quality one can get with the hand microtome.

8. LIMITATIONS

Although there are many advantages to using this method for making wood sections, there are a few limitations as well. The size of wood specimen is limited to the size of the embedding capsule, which is 8 mm (just under 5/16 in.) in diameter. Once embedded, the orientation of the specimens cannot be altered. Sections are still cut by hand, so some unevenness will remain, as compared with sections made with a rotary microtome. The method is not as fast as doing hand sections, and it is not as good as a rotary microtome.

9. ADVANTAGES

For the right application, the benefits outweigh the disadvantages.

The method is relatively quick. Embedding takes minutes, and mounting in the holder and slicing take just a few more minutes. All supplies are inexpensive and readily available. It is a reliable and easy-to-replicate process. It allows one to take smaller samples that can be useful once embedded, whereas they would be much harder to handle during conventional hand sectioning. Sections are larger, so there is less searching through multiple small sections. It is easier to compare features to a published microtomed section, as it is less obscured by air bubbles and variations in thickness. Sections are good enough to be photographed for reports or other publications.

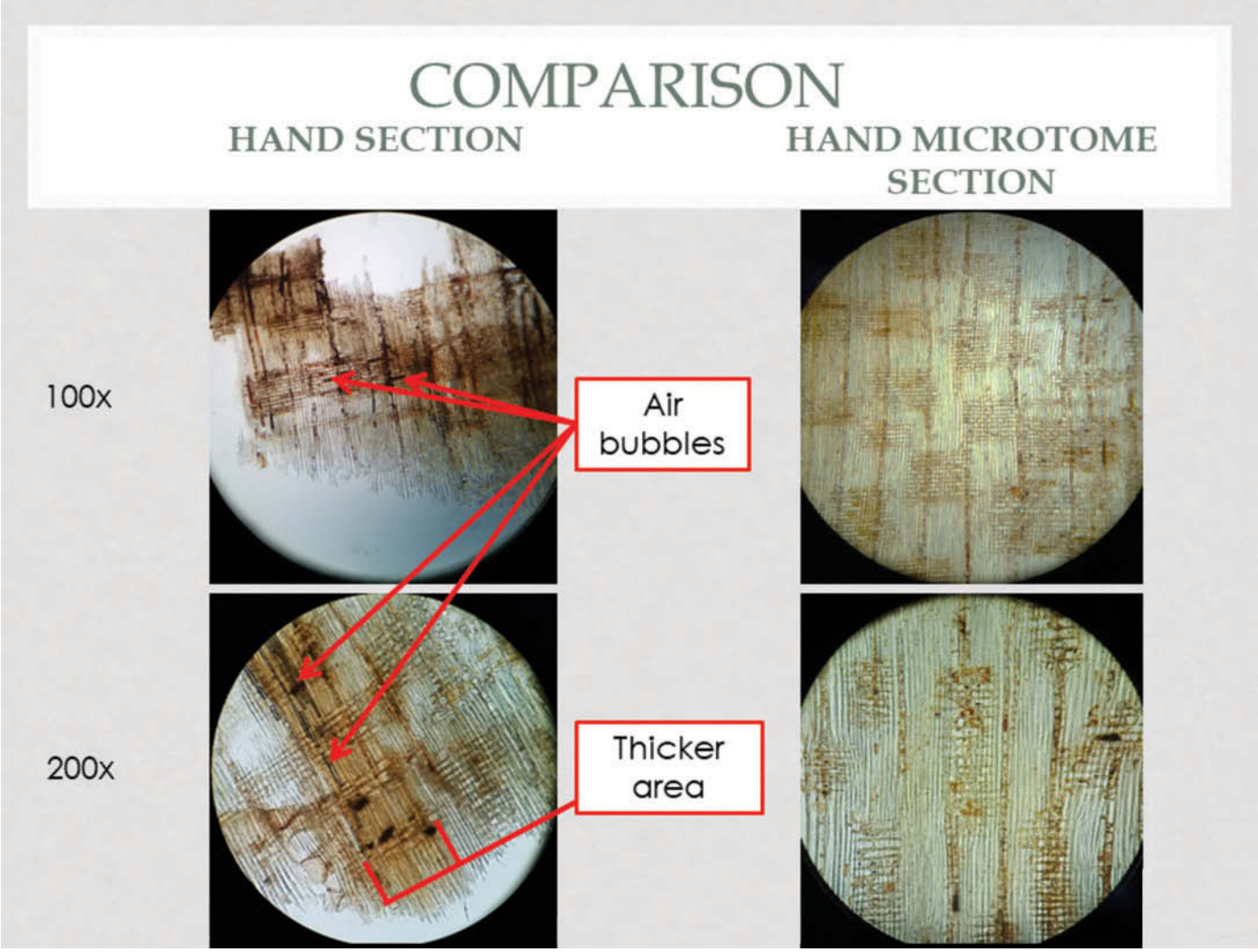

Fig. 7. Manchineel, radial, 100x at top and 200x below; cut by hand on the left and with the hand microtome on the right. Note the lack of air bubbles and more even thickness in the hand microtome sections.

Figure 7 compares a hand section to a section made with the aid of the hand microtome. Both the 100x and 200x views clearly show that the hand microtome section on the right is larger; it fills the view. It is in plane throughout the entire view. It has fewer air bubbles because it is even and thin. Finally, the images from the sections made with the hand microtome are of publishable quality.

10. CONCLUSION

This sectioning method has high potential for delivering larger and very even thin sections for wood identification and probably other identification techniques, such as textile fiber identification. It is relatively quick, easy, and inexpensive. The design of the hand microtome can be fine-tuned a little more, perhaps by having a better-fitting sleeve made by a machinist.

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Hather, Jon G. 2000. *The Identification of Northern European Woods*. London, UK: Archetype.

Hoadley, R. Bruce. 1990. *Identifying Wood*. Newtown, CT: Taunton Press.

Snedden, A. "'Tuffleye' Instructions and Tutorials." Wet a Hook Technologies website. Accessed August 27, 2019. http://www.wetahook.net/page16/page16.html.

SOURCES OF MATERIALS

- Beem Embedding Capsules, Size 00, 100/pk $8.25
  https://www.emsdiasum.com
- Climax Metals coupling RC031-S-4H, $15.50
  https://www.Amazon.com
- GEM blades, PTFE coated, $23.00 for 100 blades
  https://www.emsdiasum.com
- Mitutoyo micrometer, MHT-6.5, 148-201, $8.00
  https://www.ebay.com
- Wet a Hook Tuffleye Core, 30 cc, $21.95
  https://www.shop.wetahook.net

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DR. EDGARD O. ESPINOZA AND RANDY S. WILKINSON

Separating the Three Species of *Swietenia* spp. in Rhode Island Furniture Using Direct Analysis in Real Time–Time-of-Flight Mass Spectrometry

ABSTRACT—Separating the species of *Swietenia* spp. using traditional wood anatomy has been difficult because the cell structure of all three species is not diagnostic and the color and density overlap. Wood samples were taken from 16 pieces of 18th-century American furniture made in Rhode Island, all in the collection of the Yale University Art Gallery along with seven samples from the conservator's collection. This study compared heartwood chemotypes of 34 samples using direct analysis in real time–time-of-flight mass spectrometry to a known database. Results indicate that all three species of *Swietenia* can be reliably separated and were found in Rhode Island furniture made in the 18th century.

1. INTRODUCTION

Let's begin by looking at mahogany from a different perspective. Sometimes it is true that when you change the way you look at things, the things you look at change. We probably would all agree that the first impression of figure 1 is that we were looking at mahogany but just different pieces with slightly different colors. If given more time to study the wood, some of us would begin to question what we were looking at and maybe suggest that a couple of them are not mahogany.

Indeed, this is the case. The only mahogany piece is at the top left, as seen in figure 2. The others are (seen clockwise) muskwood, toon (sometimes called red cedar), crabwood, rose mahogany, and canjarana. All of these woods are members of the Meliaceae family or the mahogany family, but only one is a true mahogany—*Swietenia*. The other five are "mahogany look-a-likes." Therefore, a new approach is needed, a new way of seeing, and a new way to separate the mahogany species.

"Mahogany" has traditionally been described and identified differently by groups whose interests overlap but have had a different focus, namely botanists, wood anatomists, scientists, and members of the decorative arts community (curators, collectors, antique dealers, and craftsmen). Because the perspective of each group is different, the language used to identify and describe mahogany ranges from highly scientific to folklore at best. Understanding each group's perspective and the contribution each makes to identifying mahogany will ensure that the subject is not reviewed and evaluated from a single point of view.

2. BACKGROUND

To the botanist, mahogany is a member of the Meliaceae family, which contains some 52 genera and 621 species (Gasson and White 2008). The genus *Swietenia* is presently composed of three species: *Swietenia mahagoni* (Linnaeus) Jacquin, *Swietenia humilis* Zuccarini, *and Swietenia macrophylla* King. The botanist identifies its unknown by observing the flowers, fruit, leaf structure, and bark. The botanist also aids in defining the geographic area of growth. The limitation from the decorative arts point of view is that there are no flowers, fruits, and leaves of the tree; all that is left is processed wood with no information of its origins. Yet from a historical angle, proper placement of the wood into its genus and species has important implications.

The history of botanically separating the genus and species from others in the Meliaceae family is long and complicated. Before Carl Linnaeus's adoption of the binomial system of nomenclature in 1760, mahogany was classified as *Cedrela mahagoni*. Thus, for a botanist before 1760, mahogany was in the *Cedrela* genus, the same genus as Spanish cedar or cigarbox cedar. Nicolaus van Jacquin in 1760 separated *C. mahagoni* into a genus of its own, which he called *Swietenia*. It was named after Baron G. L. B. Swieten, a Dutch naturalist and physician (Keay 1996). For the next 76 years, botanically, mahogany was thought to be of this single genus and species, namely *S. mahagoni*. Between 1836 and 1837, *S. humilis*, a second species of mahogany, was described botanically by J. G. Zuccarini from specimens collected in southwestern Mexico. The final species was not added to the genus until 50 years later in 1886 by George King. *S. macrophylla* was named based on trees grown in the Botanic Garden at Calcutta from seeds apparently collected in Honduras.

In addition, it is believed that all three species grew in distinct geographic areas. The native range of *S. mahagoni* is confined to southern Florida and the West Indies. The native range of *S. humilis* grew on the Pacific coast of southwestern Mexico to Costa Rica, and the native range of *S. macrophylla* grew on the Yucatan Peninsula through Central America and into Columbia, Venezuela, Peru, and extreme western Brazil (Record and Hess 1943). It should be noted that the assignment to geographic areas of growth were largely developed in the late 19th and early 20th centuries. Keep in mind that *S. macrophylla* did not receive its own botanical name until 1886. If we were to look at a map drawn today, it would look much different owing to planting and hybridization.

Fig. 1. Mahogany—they all look similar.

Fig. 3. Transverse section of all three species of *Swietenia*.

Moving to yet another perspective, the wood anatomists have been reluctant to separate the genus into specific species because of little variation in cell structure and the wide variation in grow rate, density, and color. Figure 3 shows transverse sections or end grain sections of all three species of mahogany. Notice the color difference; notice that some of the vessels have deposits in them, some red, some white, and some not at all. The wood anatomist assigns wood to a particular genus and sometimes species by observing and measuring individual wood cells and comparing them with a known standard, sometimes can be aided by determining the specific gravity of the wood in question. In the case of mahogany, getting to the genus level (i.e., *Swietenia* spp.) is not a problem, but getting to the exact species is not so straightforward. The limitations lie in not enough clear difference microscopically and overlapping ranges of specific gravity.

In figure 4, the two pieces of wood are the same size, but have a different color and weight. Yet both are mahogany. Note the huge difference in specific gravity: *S. mahagoni* has a specific gravity of .86, whereas *S. macrophylla* has a specific gravity of .46.

Many have attempted to overcome the limitation by drawing clearer separation between species using specific gravity and cell diameter (fig. 5). Kribs (1968), in his book *Commercial Foreign Woods on the American Market*, first published in 1959, groups all three species together and generally describes the color as pale brown, pink, light red, dark red, or reddish brown. Furthermore, the wood is light and soft to hard and heavy with a specific gravity of .40 to .85. Other attempts to make the distinction between the three species relied on specific gravity, suggesting that the upper range of *S. macrophylla* does not overlap with the lower specific gravity range of *S. mahagoni* and *S. humilis* (Lamb 1966; Lane 2016). As an attempt to be more accurate with the accepted specific gravity, *S. macrophylla* was reported to have a specific gravity of .57 to .68, with the average being .62, whereas *S. humilis* had a range of .67 to .89, with an average of .76

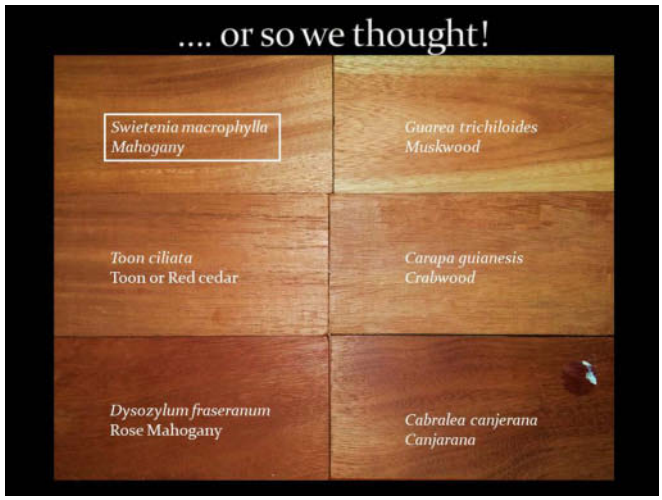

Fig. 2. *S. macrophylla* is seen at the top left.

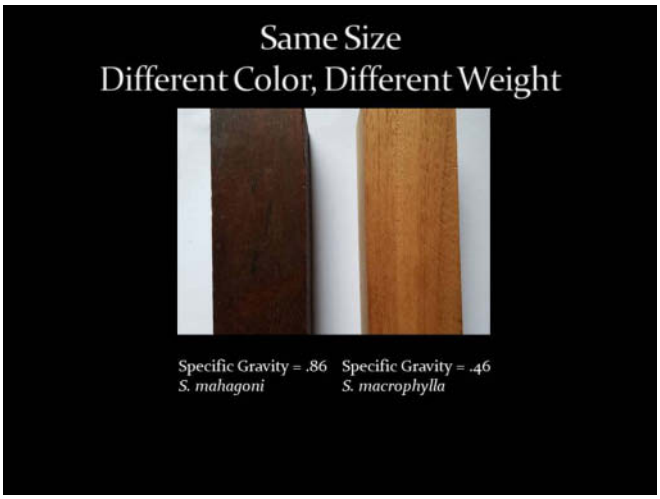

Fig. 4. Comparison of *S. mahagoni* and *S. macrophylla*.

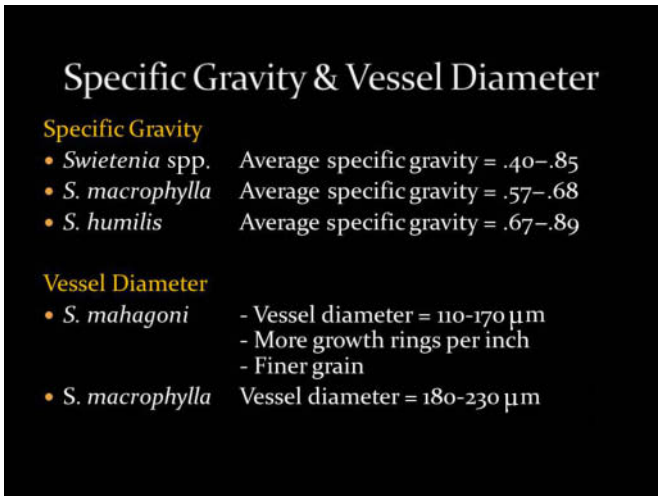

Fig. 5. Specific gravity and vessel diameter for *Swietenia* spp.

(Boone and Chudnoff 1970). A further attempt to draw some distinction between the three species relies on comparing diameters of the vessel elements and growth rings per inch, suggesting that *S. mahagoni* is slower growing, having more growth rings per inch and a finer grain (i.e., smaller vessel diameter) than that of *S. macrophylla*. It has also been suggested that the vessels are round or oval in *S. mahagoni*, having a diameter of 110 to 170 µm, whereas *S. macrophylla* has a vessel diameter of 180 to 230 µm. In the end, taking all of this information into account, the ability to reliably separate the three species can be a very well educated guess but is seldom definitive.

The members of the scientific community offer yet a different perspective. Due to the increased effort to combat illegal logging around the world, scientists have been working with wood anatomists to develop techniques to separate closely related species. Some of the promising approaches are near-infrared spectroscopy, direct analysis in real time–time-of-flight mass spectrometry (DART-TOFMS), DART Fourier transform ion cyclotron resonance mass spectrometry, DNA, pyrolysis–gas chromatography–mass spectrometry (Py-GCMS), and laser-induced breakdown spectroscopy (LIBS).

The use of near-infrared spectroscopy has been used successfully to separate *S. macrophylla* from a select group of look-a-like members of the Meliaceae family, such as crabwood, Spanish cedar, and gogo. Note that this technique cannot separate the three species of mahogany. The technique is fast and nondestructive, and this study was based on vouchered samples from 27 different countries.

DART-TOFMS has been used successfully to separate Brazilian rosewood (*Dalbergia nigra*) from other Brazilian rosewood look-a-likes. More recently, DART-TOFMS has been successful in separating the three species of mahogany to genus and species. It requires a small sample size, no sample preparation, can be minimally intrusive to the object, is very fast, and yields accurate mass measurements. It does, however, require a database of known vouchered samples by which an unknown sample is then compared. Note that this is unpublished research to date, except for the other species, such as *Dalbergia*.

Most recently, a more advanced method of DART-TOFMS was used to separate two species of *Pterocarpus*. This method required a 5-mg sample that was then made into powder and the solvent-extracted material was analyzed. Coupled with multivariate statistical analysis, it yielded a 100% accuracy rate.

The more promising science but most difficult to develop is the use of DNA. DNA extraction from plant leaf and bud tissue is fairly standard. DNA extraction for freshly harvested wood from the cambium tissue has also been found to yield DNA of high quality. But the DNA extraction from aged wood, particularly from the heartwood, is more challenging. DNA extraction technology is rapidly advancing and being used to help separate wood species and may ultimately be a key tool in wood identification.

The last perspective is that from the decorative arts. The ability to separate the three species of mahogany from the decorative arts perspective is limited at best and relies on color, density, figure, form, workability, and to some degree connoisseurship. The use of a common name and trade name compounds the problem. With at least 446 reported common names for "mahogany" used over the centuries, confusion is inherent (Alden 1998). If one factors in the language used to describe mahogany in account books, trade journals, shipping manifests, and advertisements from the 15th through 20th centuries, it becomes very clear very fast that the name is based exclusively on color, density, workability, and geographic region of growth, thus separation is based solely on connecting the geographic region of growth to density and color. For example, if an object were made of mahogany that was dark brown and dense with great figure, it was said to be "Cuban mahogany" or "Santo Domingo mahogany." What is really being said is that the wood looks like *S. mahagoni*, not the light soft mahogany *S. macrophylla*, and *S. humilis* is not even on the radar for the majority. This observation may not necessarily be incorrect, but it is not definitive. Furthermore, if one factors in the number of species that "look like mahogany" into the equation, the task to separate the three species of *Swietenia* becomes impossible given that one may he or she has an object made of mahogany, but in fact it is a look-a-like and not mahogany at all.

Reviewing books written about furniture, whether trade journals, auction house catalogs, or even books dedicated to furniture from a specific region or collection of a major museum, identification is usually done by eye. It is assumed that if the common name is used, the identification was done by eye. If the Latin name is used, it is assumed that the identification was conducted microscopically but only assigns identification to the genus level (i.e., *Swietenia* spp.).

Finally, it should be noted that not only is the focus of the aforementioned groups different, but the physical material that they are looking at is drastically different. The botanist sees the tree throughout an entire season of growth; the anatomist sees a processed piece of the tree that is cut into boards or veneer. The scientist obtains a minute sample of the wood, often needing only a few hundred micrograms of material. And finally, the members of the decorative arts community see an object that may be hundreds of years old that contains material derived from a tree that has been milled, molded, oxidized, and coated, often many times in its history.

3. SEPARATING THE SPECIES: DART-TOFMS

Now having a good understanding of past attempts to separate the species, let's turn our attention to the most current research (fig. 6).

In October 2017, 27 wood samples were taken from 16 different pieces of Rhode Island furniture in the collection of the Yale University Art Gallery. The objects to be sampled were selected by Patricia E. Kane and John Stuart Gordon. The 16 objects selected represent the pinnacle of style and craftsmanship in Rhode Island in the 18th century, and the selection was also made by observing the variation in color, grain, and quality of the woods used in these objects. Another 7 samples were taken from a private wood collection. These consisted of wood collected over the past 30 years and range from entire mahogany logs, crotch veneer, and pieces saved due to density and color.

The sample locations for all of the furniture in the collection at Yale were selected by finding the least obtrusive area to sample. This included behind locks, inside of case pieces, inside surfaces of drawer dividers, along existing damaged edges, and sites adjacent to hardware. The sample size varied from object to object based on sample location, but generally it was between 1 and 2 mm square and up to 25 mm in length.

In November 2017, the wood samples were taken to the U.S. Fish and Wildlife Forensics Laboratory in Ashland, Oregon. Working with Dr. Edgard Espinoza and his team, who developed the DART-TOFMS database for mahogany, the testing began. The advantages of DART-TOFMS are considerable. The method requires a very small sample size and no sample preparation. It can be very minimally intrusive to the object if done with care, is extremely fast, and yields accurate mass measurements (fig. 7). The first step in the process was to make tiny slivers from the collected samples and then hold them in the ion stream to obtain the data.

The database that Dr. Espinoza developed is seen in figure 8. On the top line, the average spectra for *S. mahagoni* is shown in blue. The next graph down is the average spectra for *S. macrophylla* in red, followed by average spectra for *S. humilis* in green. The finial graph in yellow is the atypical chemotype spectra for what is *S. mahagoni* or *S. humilis.* The key information to observe is that there are two chemotypes for *Swietenia*. One that has a mass/charge data above 760 represented in blue, red, and green, and one that does not is represented in yellow.

To add further clarity, kernel discriminant analysis was used to place the data in a more readable form in which all four groups clearly separate on a three-dimensional graph (fig. 9). The dark blue dots represent *S. mahagoni*, the red dots represent *S. macrophylla*, the green dots represent *S. humilis*, and the light blue dots represent the atypical chemotype suggesting either *S. mahagoni* or *S. humilis*. One last point to note here is that this model has a 92% accuracy rate. Figure 10 is a graph with the Yale samples added to it as black dots. The samples clearly separate into one of the four clusters. On this particular graph, six of the samples taken from the furniture are represented: one identified as *S. mahagoni*, two as *S. macrophylla*, two as *S. humilis*, and one as either *S. mahagoni* or *S. humilis*.

Fig. 6. The research project.

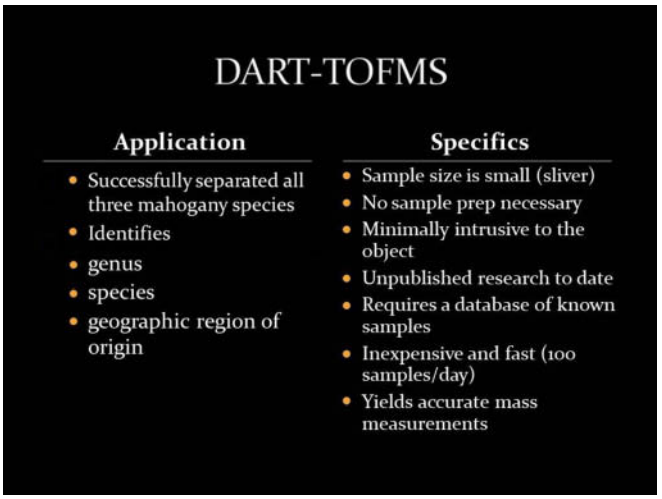

Fig. 7. DART-TOFMS.

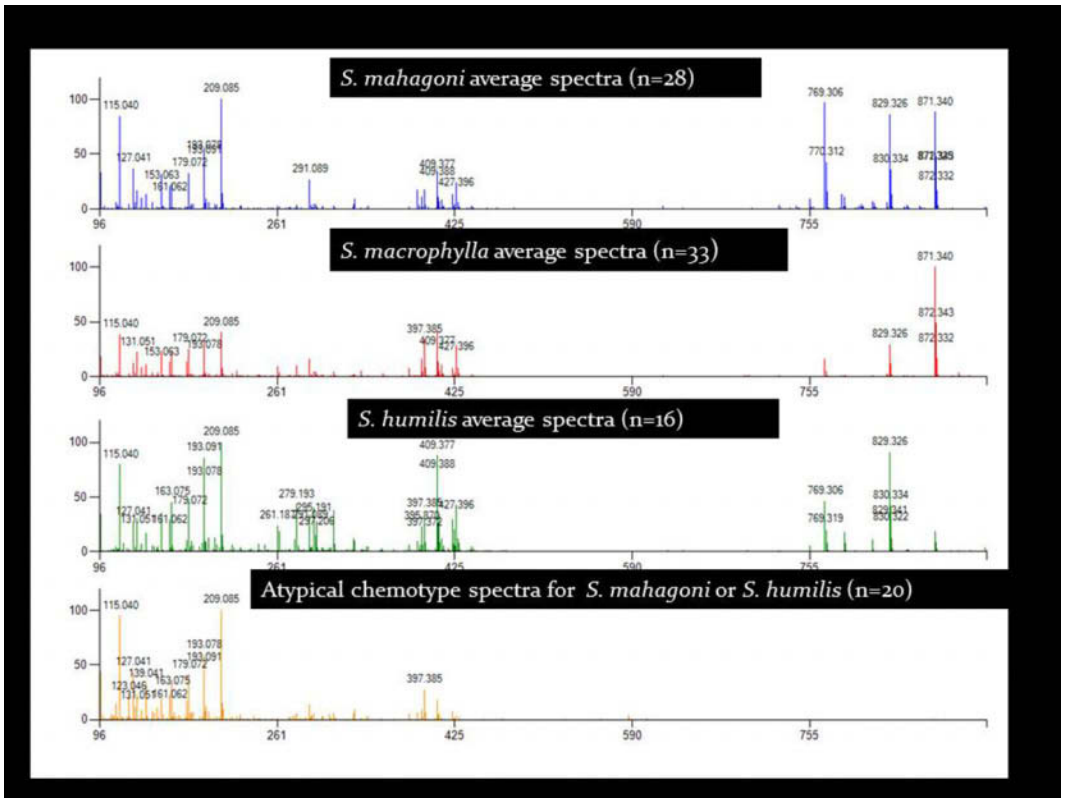

Fig. 8. Average spectra for all three species of *Swietenia*.

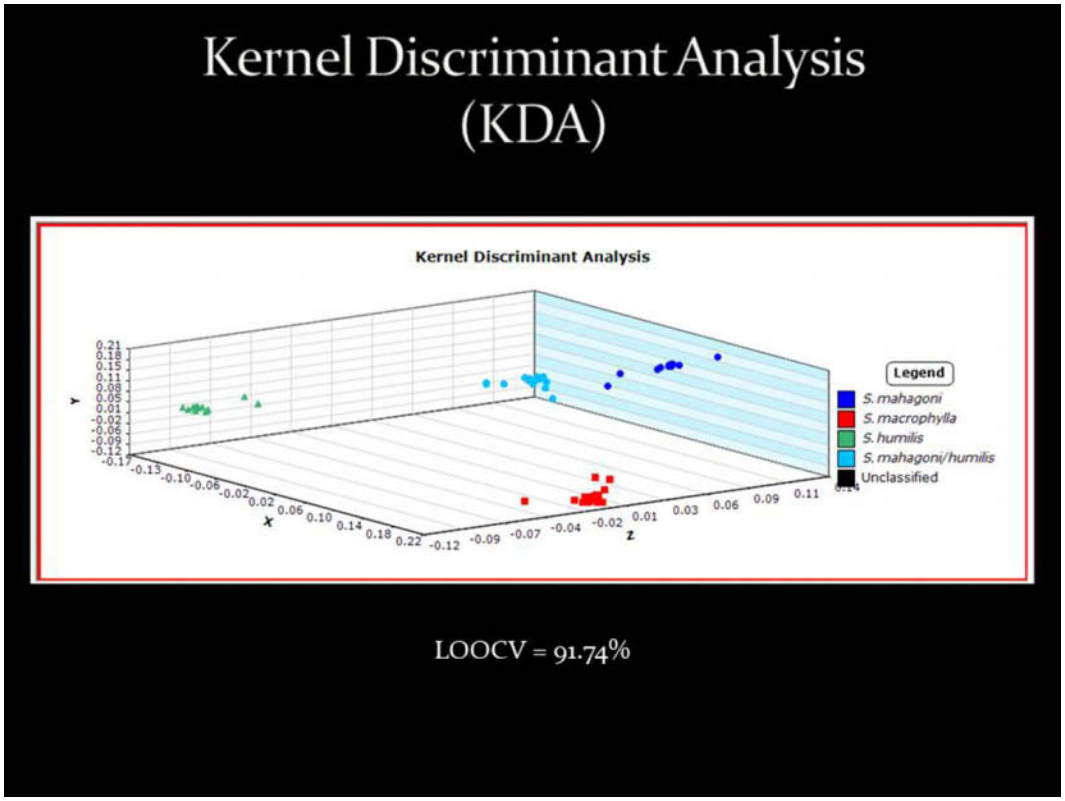

Fig. 9. Kernel discriminant analysis of the average spectra of *Swietenia*.

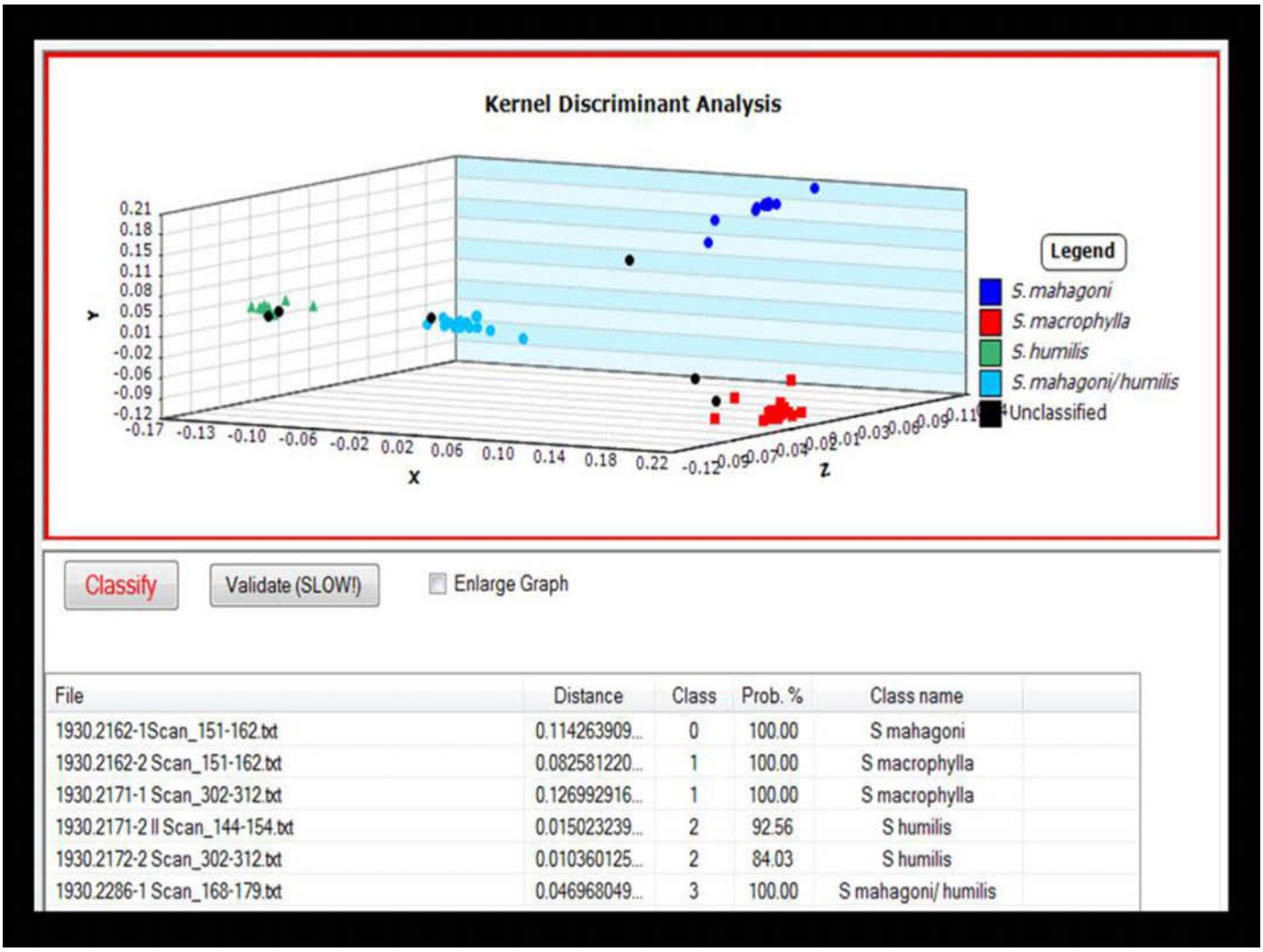

Fig. 10. Kernel discriminant analysis with six of the Yale samples represented as black dots.

4. RESULTS

The results of the analysis suggest that 10 samples were identified as *S. mahagoni*: 9 from the furniture at Yale and 1 from the private wood collection. Seven samples were identified as *S. humilis*: 4 from the furniture at Yale and 3 from the private wood collection. Four samples were identified as *S. macrophylla*: all from samples taken from Yale. Twelve samples were identified as either *S. mahagoni* or *S. humilis*: 9 taken from the furniture at Yale and 3 from the private wood collection. Finally, 1 sample from Yale was only identifiable to the genus level (fig. 11).

Now follow three brief case studies of the objects that were sampled to demonstrate in concrete terms what this research revealed. The first object was a wonderful Rhode Island chest-on-chest, as seen in figure 12. A sample was taken from the back of a drawer front, behind the lock, and from the proper right side along an existing defect. Notice that the drawer front was made from *S. mahagoni* and the case side from *S. macrophylla*.

The next example was that of a great Townsend high chest, as seen in figure 13. A sample was taken from the back of the drawer by removing the lock and then taking the sample, and the second sample was taken from the proper right side of the upper case side, near the back where a nail from the backboard caused a split. Notice that *S. humilis* was used on the front of the chest and *S. mahagoni* on the side.

The final example was from the fabulous chest of drawers seen in figure 14. Two samples were taken from the chest: the first from the underside of the drawer front and the second from the back edge of the proper right case side. Notice again that two different species were used to construct the chest; *S. mahagoni* was used to construct the drawer front and *S. macrophylla* was used for the case side.

Although one phase of the work was completed, the research did not end there. In September 2018, all 34 samples were taken to the U.S. Forest Products Lab in Madison, Wisconsin for

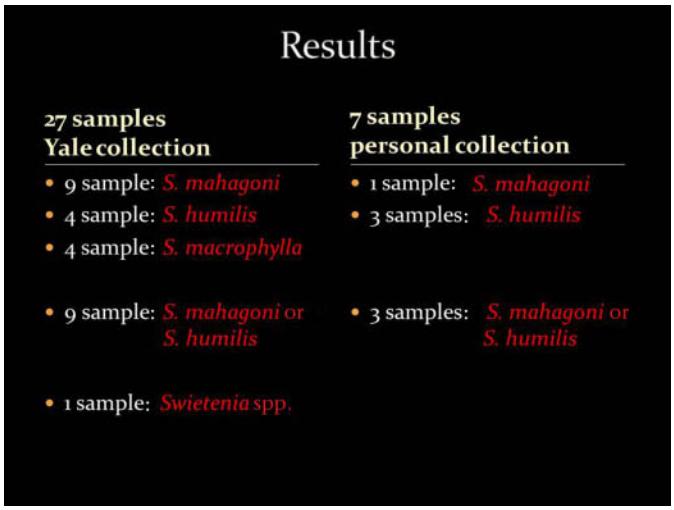

Fig. 11. DART-TOFMS results for all 34 samples tested.

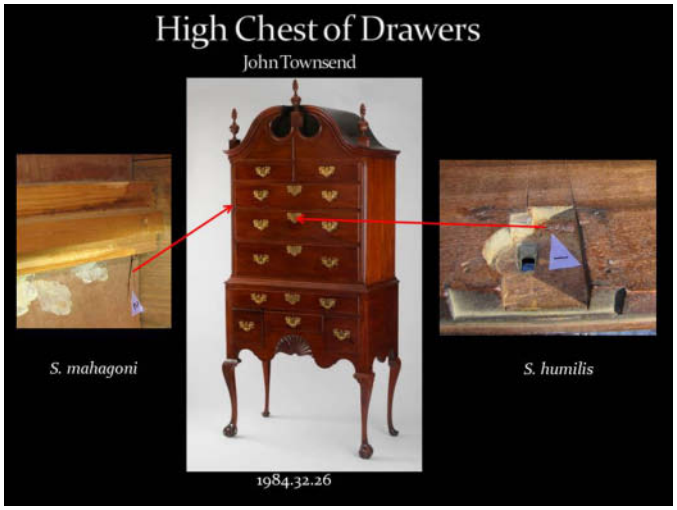

Fig. 13. High chest of drawers, John Townsend 1984.32.26. Courtesy of the Yale University Art Gallery.

further documentation. The goal of this research was to obtain photomicrographs of all 34 samples. Now there are photomicrographs of the three principal planes—transverse, radial, and tangential—for each sample, along with measurements for key cell types (fig. 15). In addition, 53 samples of mahogany were brought back to Yale for further testing via Py-GCMS.

5. CONCLUSION

The research suggests that DART-TOFMS was successful in identifying the presence of all three species of mahogany in the furniture of Rhode Island of the 18th century. The study also confirms that different species of mahogany were used on the same piece of furniture. *S. macrophylla* was found in only 4 of the 16 pieces of furniture from which samples were taken.

As with all cutting-edge research, there are unanswered questions. The first is this: What is the reason for the atypical chemotype? Is it purely biological? Is it a function of our sample site (i.e., where the sample was taken from, close to the pith of the tree, or closer to the bark? Can we further differentiate this second chemotype to suggest the positive presence of *S. mahagoni* or *S. humilis*? Keep in mind that 12 of the 34 samples shared this atypical chemotype. What if all 12 suggest *S. humilis*? Would that cause us to rethink the accepted geographic area where *S. humilis* grew?

Therefore, if *S. humilis* were not a commercially viable species, why is it found in the furniture of the 18th century, particularly Rhode Island furniture? After all, it was reported to only grow on the west coast of Central America. How did if find its way to Newport, and almost as important, why does *S. humilis* occur in large boards, planks, and veneers in a more modern collection?

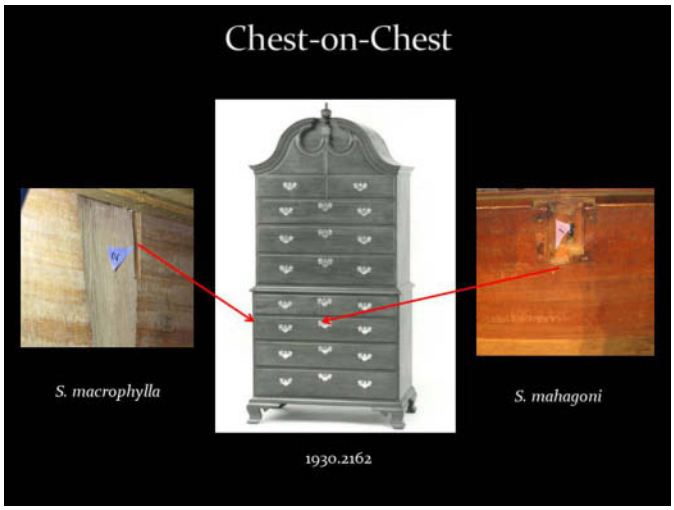

Fig. 12. Rhode Island chest-on-chest 1930.2162. Courtesy of the Yale University Art Gallery.

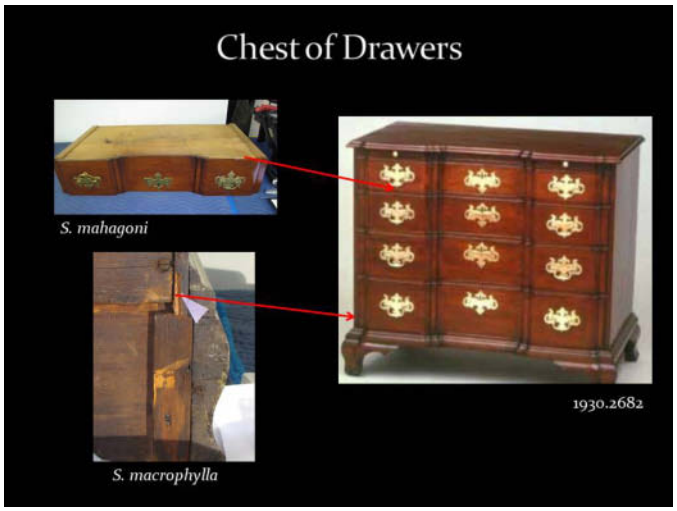

Fig. 14. Chest of drawers 1930.2682. Courtesy of the Yale University Art Gallery.

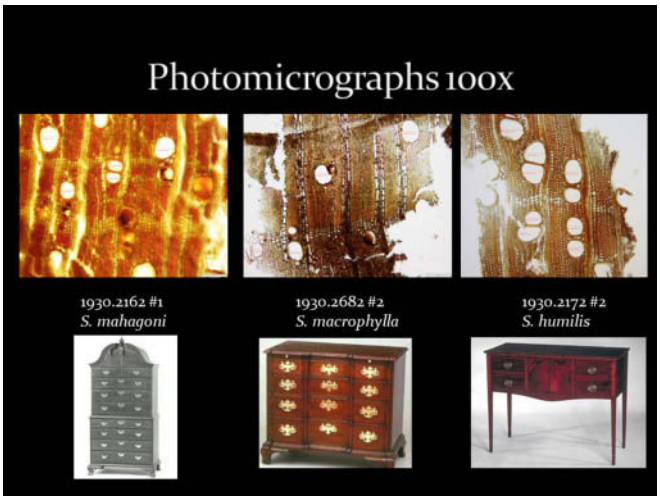

Fig. 15. Select photomicrographs, transverse section, 100x. Furniture photographs courtesy of the Yale University Art Gallery.

Considering that these important questions remain, the team is working to find answers to these questions using alternative methods, specifically the use of laser-induced breakdown spectroscopy (LIBS) and Py-GCMS. The team is actively collecting vouchered samples to build a database of both the three species of mahogany and the mahogany look-a-likes. Another grant from the Wunsch Americana Foundation was obtained to collect material from the mahogany look-a-likes and perform LIBS testing on these samples. Maybe even more exciting, actual core samples from *S. humilis* were obtained directly from Mexico. With the help of Dr. Marcelo Pace, it is now possible to sample these specimens by DART-TOFMS, Py-GCMS, and LIBS at known increments from the cambium to the pith. This is critical and may be key to understanding the two different chemotypes seen in mahogany. These scientific tools may hold the means to answering not only the remaining chemical questions but also building a database that can be shared by all institutions.

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Wooden Artifacts Group Session

Analysis of Black Resin of a Late Period Coffin by Gas Chromatography-Mass Spectrometry

Abdelmoniem Mohammed, Naglaa Mahmoud, and Wael S. Mohamed

The present study focuses on black resin's composition, beginning and uses. Black resin was used to cover funerary furniture like coffins, shabti statues and boxes, stelae, canopic chests, human and animal statues, and statue bases. The study utilized gas chromatography-mass spectrometry (GC-MS) to analyze black resin. Black resin is composed of natural resins like mastic, colophony, beeswax, bitumen, and an unknown compound. Natural resin is reported to contain essential oil. Some inscriptions on the tomb of Thebes in Egypt named it sntr, and mastic resin was of high value in Ancient Egypt. Black resin had anti-fungi and antibacterial properties, as well as insect repellents. The sample was taken from a coffin dating back to the Late period to analysis it to know it's composition to choose the best material for consolidation. The coffin, under investigation, was covered internally with a layer of black resin.

Characterizing Asian Lacquer Surfaces Using Surface Metrology and Multimodal Imaging Techniques: A New Approach

Marianne Webb, Patrick Ravines, Jiuan Jiuan Chen, and David Sheets

In preparation for the Getty Conservation Institute's Asian lacquer cleaning project, 15 different formulas of Asian lacquer were prepared using laccol, thitsi and urushi. The formulas within the three lacquer categories each differ from the next in the series by one ingredient. This way we will be able to understand how each ingredient affects the behaviour of the surface. Observation and examination of the surface at each stage of the experiment is key to following the changes over time. The Asian lacquer panels were prepared during 2017, by Marianne Webb and Sunhwa Kim, Art and Design Department at Buffalo State College, according to strict protocols to limit differences and ensure standardization of the final products. The three types of Asian lacquer, urushi, laccol and thitsi were obtained from reliable sources. Five formulas of each type of lacquer were produced and all stages were made using the same type of Asian lacquer. Each Koskisen plywood panel was sealed with raw lacquer, and then a ground coat of tonoko and raw lacquer was applied. In the case of thitsi lacquer, bone ash was also incorporated in the formula. Ground coats were polished smooth and sealed with the same lacquer. Test formulas were applied by different means. Urushi and laccol lacquers were applied by brush, however, due to the high viscosity thitsi was applied with a silicone spatula or squeegee. With exception of the roiro urushi none of the coatings were polished after drying.

Multimodal imaging: All the samples were documented with different photographic techniques with a modified UV-VIS-IR DSLR camera. Reflected IR and IR-induced IR luminescence techniques were particularly useful in revealing the differences among the different Asian lacquer panels. Surface metrology and multi-scale analysis of the Asian lacquer panels will be introduced and discussed. All 15 panels were investigated using confocal microscopy: Each lacquer panel was examined at 12 distinct areas of interest using a 10x (area 1,600x1,600 μm) and 50x (320x320 μm) objectives. Each magnification shows different physical features to consider. Surface texture can be described by the data reduction techniques of amplitude (height) parameters and spatial parameters. Physical lateral surface features such as peaks and pits and other features at each magnification are also invoked since they are not considered by both amplitude and spatial parameters. The above will be presented in hopes of starting a discussion based on: what identifying features are of interest? Are the features chosen at these magnifications good to define lacquer surfaces? Are the features at these two different magnifications related or relatable in any way? And more.

Art Shapes: An Investigation of Hans Arp's Constellations II

Madeline Corona, Dr. Georgina M. Rayner, Melissa Venator, and Angela Chang

Constellations II is a thirteen-panel, wooden wall relief designed by Hans Arp (also known as Jean Arp, 1886–1966) for Harvard University's Graduate Center in 1950. One of several artworks commissioned by Walter Gropius and The Architect's Collaborative for Harvard's first modernist building on campus, the relief is unique in Arp's oeuvre as his only large-scale wood relief and his first

architectural commission. To prepare for the relief's display in conjunction with the upcoming exhibition The Bauhaus and Harvard at the Harvard Art Museums, a technical study was undertaken to better understand its condition history and earlier surface appearances, which directly informed treatment and interpretation. Constellations II is a complex case study that offers insight into the working relationship of two prominent art historical figures as well as the challenges of treating a work that was never fully resolved and was removed from its original context. Installed in the Graduate Center's dining room in 1950 with the title Constellations, the relief originally had a dark red "natural" finish that showcased the graining of the redwood forms. Archival records, photographs, and correspondence between Arp and Gropius indicate that the artist sent instructions to rearrange and modify the relief in 1958 in order to protect the panels from damage and account for viewing obstructions in the room. As part of the revised design scheme, Arp also requested that the panels be painted blue—a compromise between his evolving thoughts on the relief and Harvard's limited budget for the adjustments. Even after these major alterations prompted the renaming of the relief to Constellations II, drastic changes continued to affect its appearance. By 1975, a series of undocumented painting, stripping, and coating campaigns had taken the appearance from blue, to white, and back to red, leaving the surfaces scratched, patchy, and uneven. Records of these campaigns survive only in sporadic photographs from the 1950s to the 1980s and on the relief itself, where remnants of paint and coatings are present on the edges, backs, and recesses of the panels. It is unclear whether these later modifications were sanctioned by either Arp or Gropius, who both died in the late 1960s, and the motivations behind them are completely unknown. In 2004, the relief was deinstalled from the dining room as part of a larger renovation project and transferred to the care of the Harvard Art Museums. Scientific analysis of the paint and coating layers on the panels helped define a timeline of alterations and corroborate surviving archival documentation, allowing the project team to make an informed decision to return the panels to their original dark red appearance. A digital tool was created to share the past iterations of the relief with the public and to better explain its history at Harvard University. This approach was deemed to be the best compromise to present the relief with an exhibitable surface while respecting the object's history and the artist's statements about his work.