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Supplementary issue paper

Some observations on the composition of Chinese lacquer

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This paper summarizes the various information that has been gathered in recent years at the J. Paul Getty Museum and Getty Conservation Institute with regard to the organic constituents of Chinese lacquer formulations. While this summary of materials is by no means comprehensive or complete, it captures the current state of the authors' knowledge, with the information itself and the bibliography intended to serve as a useful foundation from which further research may proceed. Considerable advances have been made in the last decade in the technique of pyrolysis-gas chromatography–mass spectrometry with thermally assisted hydrolysis and methylation using tetramethylammonium hydroxide and in subsequent data interpretation methodologies. These have dramatically improved the sensitivity and specificity of organic analysis. Perhaps equally important, these methodologies are allowing researchers to collaborate much more effectively and to share results that are truly comparable and reproducible. As more researchers participate and collaborate, and as more results are aggregated into shared databases, significant new insights into the methods and materials of Chinese lacquer are sure to follow.

Keywords: Chinese lacquer, Py-GC–MS, Laccol, Urushiol, Cedar oil

Introduction

Background

The collection of the J. Paul Getty Museum (JPGM) includes several pieces of French furniture dating to the mid-eighteenth century that incorporate panels of Asian lacquer as part of their surface decoration. In 2006, conservators at the JPGM and scientists at the Getty Conservation Institute (GCI) began a collaborative project to study these objects and to develop an improved analytical procedure that would allow the simultaneous analysis of the organic composition of Asian and European lacquer materials. By 2008 a preliminary literature survey had been completed, a core set of reference materials had been assembled, and an improved procedure using thermally assisted hydrolysis and methylation with tetramethylammonium hydroxide (TMAH) for pyrolysis-gas chromatography–mass spectrometry (THM-Py-GC–MS) was presented (Heginbotham *et al.*, 2008). It soon became apparent that the new method was revealing an unexpected complexity in historic Asian lacquer formulations, as a wide range of unexpected

and previously unreported materials were detected in lacquer in the JPGM collections. Our attention focused first on the surprising composition of seventeenth-century Japanese export lacquers, and a summary of our results regarding these materials was first presented in 2009 (Heginbotham & Schilling, 2011).

With the potential of the new analytical protocol established in our minds, we next concentrated our efforts on streamlining our procedures for data interpretation and on expanding the range of materials and formulations in our reference collection. In parallel with this work, we began to conduct analysis for and with an increasingly large pool of collaborators in museums and research institutions around the world. Through contributions from our collaborators we produced a significantly expanded library of chemical compounds, identifiable with THM-Py-GC–MS, that are critical to identifying the raw materials present in Asian lacquer samples.

Increasing interest in our research led to the development of a GCI workshop entitled 'Recent Advances In Characterizing Asian Lacquers' (RADICAL), through which we have shared our sampling, staining, and data interpretation methods,

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as well as the library of marker compounds that we have compiled. Venues for RAdICAL workshops have included the Getty, Los Angeles, USA, (2012), the Institute for Preservation of Cultural Heritage at Yale, New Haven, USA, (2013) and the Center for Research and Restoration of the Museums of France, Paris, France (2014).

Chinese lacquer

In the years since we began the method development phase of our research on Asian lacquer, we have had the good fortune to be able to study, sample, and analyze lacquer artifacts from across Asia and Southeast Asia. In particular, we have studied a considerable number of Chinese artifacts from as long ago as the second century BCE and have found many compounds present in these samples that were, at first, mysterious to us. As was the case with our study of Japanese export lacquer materials, each discovery of some group of new compounds in the results from THM-Py-GC-MS would lead to further research to try to understand their possible origins in the variety of raw materials that have found their way into lacquer formulations across China and through the years. Often, an interpretation of the chemical structure of the compounds would point to some possible classes of raw materials. Then, a search of the historical literature for recipes or other references materials would refine the possibilities. In addition, inquiries directed to lacquer craftsmen helped us to understand the function of different additives in lacquer formulations, while further searching of the scientific literature might connect our findings to research that had been conducted on these compounds or raw materials in allied fields.

This paper is intended to summarize the various information we have gathered over the years and the trends we have observed with regard to the organic constituents of Chinese lacquer formulations. We are fully aware that this material is by no means comprehensive or complete; it merely represents the state of our knowledge at the moment. Still, we hope that this information and the references compiled here will provide a useful foundation from which further research may proceed. Considerable advances have been made in the last decade with THM-Py-GC-MS and in subsequent data interpretation methodologies that have dramatically improved the sensitivity and specificity of organic analysis (Schilling *et al.*, 2016). Perhaps equally important, these methodologies are allowing researchers to collaborate much more effectively and to share results that are truly comparable and reproducible. As more researchers participate and collaborate, and as more results are aggregated into shared databases, significant new insights into

the methods and materials of Chinese lacquer are sure to follow.

Anacards

It had long been assumed that Chinese lacquerware has always been made from the urushiol-based exudate of *Toxicodendron verniciflua* trees that grow throughout specific regions of China, Japan, and Korea. However, numerous analyses conducted at the Getty since 2007 have shown that many Chinese lacquer objects produced for export in the early eighteenth century were formulated with laccol-based lacquer, which is the exudate from *Toxicodendron succedaneum*. It is generally considered that these export lacquerwares were produced in southern China. This corresponds well with the identification of laccol-based lacquer in a group of eighteenth- and nineteenth-century Cantonese objects in Portuguese Royal Collections (Petisca *et al.*, 2011). Although *T. succedaneum* is traditionally associated with Vietnamese and Taiwanese lacquers (Nimura & Miyakoshi, 2003), much Chinese export lacquer was produced in the south of China, near regions where this tree is widespread. Recent taxonomic research suggests that the range of so-called Vietnamese lacquer may extend further into southern China than was previously known, with laccol trees identified in Guangxi Province (Wan *et al.*, 2007).

A substantial number of Chinese lacquer objects made for the domestic Chinese market over the course of the last 2000+ years have also been analyzed at the Getty. As expected, most were found to have been made using urushiol-based lacquer; however, this was not always the case. One unusual example was a mid-sixteenth-century filled-in polychrome lacquer dish from the Victoria and Albert Museum, London, UK, (FE.87–1974), thought to have been made in northern China and which depicts a five-clawed dragon design and bears the mark of Emperor Jiajing (1521–66 CE). The ground (or foundation) layers were found to have been made with laccol-based lacquer (with additions of cedar oil and camphor), whereas a mixture of laccol and urushiol was identified in the upper red layers. This may be explained by parallel archival research into contemporary Ming Dynasty records, which state that large quantities of laccol from a region that is within present-day northern Vietnam were offered to the Chinese Imperial court as tributary gifts (Huang, 1962). This implies that laccol was available to Chinese craftsmen working even in the north of China, confounding the conventional wisdom regarding lacquer use and availability.

One unusual finding with respect to the laccol-based lacquers that were analyzed was the detection of significant levels of polysaccharides within the laccol-

containing layers. Although catechol-rich saps exuded by lacquer-producing trees contain small amounts of naturally occurring glycoproteins and carbohydrates (Kumanotani, 1998), these components have never been reported in Py-GC–MS studies of underivatized *urushi* and laccol lacquers (Lu *et al.*, 2007). In THM-Py-GC–MS of urushiol-based lacquers, methylated carbohydrate monomers and dimers are barely detectable, even with the aid of selected ion chromatograms (SICs). In contrast, every laccol-containing sample that was analyzed (including several modern reference materials) showed two prominent series of peaks easily located with the SIC for mass 129, and their mass spectra corresponded most closely to various methylated derivatives of uronic acids. This finding is in accord with research on raw lacquer compositions undertaken in Japan in the 1990s, which demonstrated that the carbohydrate content of laccol sap is nearly three times higher than that of *urushi* sap (Kumanotani, 1995). Undetectable by Py-GC–MS without the use of TMAH, these markers have not been reported in analytical studies of lacquered objects prior to our research.

In peak area reports for the eighteenth-century Ryukyuan inkstand from the Getty collection, the laccol carbohydrates made up a remarkably large fraction of the total lacquer sample, totaling more than 10% of the combined peak area of all detectable species. These compounds are highly unlikely to have originated from a lacquer additive, as they were also detected in fresh sap from a *T. succedaneum* tree tapped by the authors. The exact distribution and function of the carbohydrate fraction in cured Anacard lacquer films is still not well understood, although several possible models have been proposed (Lu & Miyakoshi, 2015, pp. 75–88; Keneghan, 2011). The discovery of significantly elevated carbohydrate contents in laccol-based lacquers highlights the importance of better understanding the role of polysaccharides in the light-induced deterioration of lacquer and the possible implications for preventive and interventive conservation that follow.

Oils

Systematic review of the Chinese literature on the topic of lacquer formulations makes it clear that a wide range of vegetable oils has been added to *qi* lacquer by Chinese artists over the course of thousands of years. The identification of oils in samples taken from historic objects is a complex proposition and is discussed at length by Schilling *et al.* (2016). To summarize, certain indications regarding oil identification may be garnered from the profile of fatty acids detected by Py-GC–MS. For instance, oils that are classified as drying oils typically yield high ratios of

azeleic acid to palmitic acid methyl esters (the so-called A/P ratio). Certain oils will produce characteristic ratios of palmitic to stearic acid methyl esters (P/S ratios); notably, tung oil (derived from *Vernicia fordii*) typically yields a P/S ratio of 1.0–1.2 which is uniquely low among the oils known to have been used in lacquer production. As another example, the presence of a certain class of compounds known as methyl alkylphenyl alkanoates (APAs), which are formed from highly unsaturated linolenic acid and eleostearic acid by bodying linseed, perilla or tung oils at elevated temperatures (Evershed *et al.*, 2008), can point to the presence of these processed oils.

Unfortunately, an additional complication arises in the interpretation of fatty acid profiles due to the fact that, besides oils, a number of other materials used in the production of lacquer also contain fatty acids, including Anacard lacquers themselves. Even in the best of circumstances, partial overlap in P/S ranges makes it impossible to differentiate unambiguously between perilla oil, sesame oil, tallow tree oil, and linseed oil. The possible mixing of oils confuses the situation and, additionally, it can be difficult to assess the extent of influence of fatty acids from *non-oil* sources on the profiles from analyzed sample material, further complicating interpretation. For a deeper discussion of the characterization of oils using THM-Py-GC–MS, see Schilling *et al.* (2016).

The five most important drying oils associated with the production of Chinese lacquer seem to be perilla, tung, sesame, tallow tree, and linseed. The dates of use of these oils are difficult to deduce from the historical Chinese literature: for a fuller discussion of the subject, see Chang and Schilling (2016). In general, it would appear that perilla and tung oils have been the most widely used over the longest period of time. These oils are not always clearly differentiated in ancient Chinese texts.

In our investigations of Chinese lacquered objects to date, we have invariably found oil to be a significant component of the lacquer layers, although not always of the ground layers. In general, it seems that oil is added to Chinese lacquer formulations to a somewhat greater degree than it is in Japanese lacquered objects. The addition of oil to lacquer has a number of effects on its cost, working properties and on the characteristics of the cured film. Oils are invariably less expensive than lacquer so their addition may be motivated by reasons of economy. The addition of oils can also make liquid lacquer less viscous so that it is more easily brushable, and improve its leveling. With regard to cured-film properties, the addition of oils generally increases the gloss of the lacquer film and can also render a lacquer film more transparent, softer and slower to cure.

Carved lacquer

It is interesting to note that Chinese craftsmen who produced carved lacquer may have taken conscious advantage of the softness and slower curing of lacquers mixed with oils. Lacquer that is to be carved must not be so hard and brittle that it is prone to chipping and breakage during carving. Because lacquer can continue to polymerize over a period of weeks and months after initial curing (Webb, 2000, p. 8), it can become progressively harder and more brittle over this timescale. From the carver's point of view then, it is critical that the prepared lacquer substrate not harden too much before the carving is complete or else the work will be ruined. It would seem that a traditional manner of preventing premature hardening has been the addition of significantly more oil to the lacquer than is customary in flat lacquer production. To date, we have only been able to analyze samples from seven objects of Chinese carved lacquer, dating from the fifteenth to eighteenth centuries. These objects include a variety of types, from Ming imperial ware to an eighteenth-century bowl from Yunnan to export ware from the southeast. Nevertheless, each of these pieces has in common a very high proportion of oil in the formulation. In most cases, the combined fatty acid peak area comprises more than 90% of the sum of all analyzed compounds while the Anacard components typically total only 3–7% of peak area. These proportions are quite unusual in flat (uncarved) lacquer that the authors have analyzed.

Perilla oil

The earliest citations found for the addition of oil to lacquer go back to the later part of the Han Dynasty, from 206 BCE to 220 CE (Tao & Shang, 1986) and appear to refer to perilla oil, which is pressed from the seeds of *Perilla frutescens*. Many believe that perilla oil was the dominant oil used in early times (Huang et al., 2004), although some scholars suggest that it may have been confused with tung oil in early texts (Cheng & Zhang, 1991). Our studies of Chinese lacquer objects have also very commonly found oil additions to upper, glossy lacquer layers whose fatty acid profiles show P/S ratios in the range 2–4 and high A/P ratios. Based on the available literature, this type of oil would seem most likely to be perilla oil. This is particularly common in southern Chinese export lacquer of the eighteenth century, but it appears to be widespread in Chinese lacquer from a broad range of sources. Unfortunately, perilla oil is very difficult to distinguish analytically from tallow tree or linseed oil.

Tung oil

The earliest clear mention of tung oil in association with lacquer that we have found in the Chinese

literature dates to the Southern Song Dynasty, from CE 1123 to 95 (Cheng & Zhang, 1991), although its use would seem to go back much further. Tung oil is derived from the seeds of *Vernicia fordii*. In the course of our analytical program, one of the oldest lacquer artifacts that we analyzed, an archaeologically recovered Western Han Dynasty ear cup from Mawangdui, yielded a fatty acid profile with a very low P/S ratio that is almost certainly indicative of the use of tung oil in the formulation. Our analytical research has found significant amounts of cold-pressed and heat-bodied tung oil in all periods studied. Interestingly, we have also found numerous examples of tung oil that contain significant amounts of APAs, suggesting that the oil underwent significant anoxic heat bodying prior to use (Evershed et al., 2008).

The earliest reference we have found to the heat bodying of tung oil in the Chinese literature is from a famous architecture treatise of the Northern Song Dynasty, *Yingzao Fashi (Treatise on Architectural Methods)* completed in 1110 CE and published in 1103. The writer Li Jie, who was a distinguished architect and an official in the Directorate of Construction, was appointed by Emperor Zhezong to compile previous works into an official court manual and set standards for architects' constructions. The document calls for 'decoction' of tung oil using both low and high flame fire (Li, 2006). The heat bodying of tung oil is a very delicate proposition; the oil is so highly unsaturated, and thus prone to polymerization, that heat bodying can, if not carefully controlled, easily lead to the coagulation of the oil into an unusable jelly. As it is not yet clear how the addition of heat-bodied tung oil affects the working properties or cured-film properties of lacquer, we cannot speculate on the reasons that craftsmen would have added this material. It is interesting to note, however, that the APAs formed during the anoxic heat bodying of tung oil are less reactive than their straight-chain precursors and so might behave more as plasticizers than as polymer-forming compounds in the cured lacquer. Further research into the properties and usage of heat-bodied tung oil in lacquer would certainly be beneficial.

Tallow tree oil

Chinese tallow tree oil, a drying oil also referred to as stillingia oil, is pressed from the seeds of *Sapium sebiferum* (recently renamed *Triadica sebifera*). As the tree's main distribution is in southern China, the oil's production areas include: Zhejiang, Jiangxi, Guangxi, Hunan, and Hube provinces. Interestingly, the above provinces are all known for their lacquer production. This oil is noted extensively in Chinese sources for its excellent drying properties (Song

et al., 1966), attributed to high concentrations of 2,4-decadienoic acid and 8-hydroxy-5,6-octadienoic acid, which are unusual fatty acids not present in other oils (Chen *et al.*, 1987). Because of difficulties in obtaining pure Chinese tallow tree oil in our research, we were unable to confirm the fatty acid compositions given in the references. Given its unusual fatty acid composition, it may eventually be possible to identify specific pyrolysate markers in lacquers made with this oil.

Linseed oil

Linseed oil, which is the dominant drying oil of the western world, is pressed from the seeds of the flax plant *Linum usitatissimum*. In China, the history of its use is not entirely clear from our survey of the literature, in part because during some periods the Chinese name for flax seed can be easily confused with that for sesame (Qiao, 2004). The occurrence of flax seed in China was first mentioned in a few documents that date to the Han Dynasty. At that time, the distinction between sesame and flax seeds was still clear, but a dictionary written in the third century CE, in which sesame and flax were combined into one plant, may have triggered centuries of confusion. Linseed oil is mentioned unambiguously in the relatively modern Chinese literature of the seventeenth century (Needham *et al.*, 1996, pp. 7–11; Song *et al.*, 1966), although not in the context of lacquer production. Rein (1889, p. 155), writing in the late nineteenth century, states that linseed oil was unknown in East Asia until ‘recent times’. Whatever the case, it seems that linseed oil is likely to have played only a very minor role, if any, in historical Chinese lacquer production.

Sesame oil

Sesame oil is a non-drying (or poorly drying) oil that seems to be only rarely associated with lacquer making. The earliest reasonably certain references that we have found in association with lacquer go back to the Northern Song Dynasty, CE 960–1127 (Anon., 2001). Literature that refers to the use of sesame oil in lacquer production normally specifies its use in either polishing or cleaning lacquer and not as an additive to the lacquer itself. One of the Northern Song recipes does, however, call for adding sesame oil to retard curing, so that the lacquer does not dry too quickly. In addition, Bonanni (2009, p. 23), writing in the early eighteenth century very specifically says that cooked sesame oil was added to lacquer in China, citing as his source a Portuguese friend who had lived in China and observed lacquering on several occasions. Based on its fatty acid profile, sesame oil should be reasonably distinctive in the results from THM-Py-GC/MS (Schilling *et al.*, 2016), although, we have yet

to find any indication of its use in Chinese lacquer that we have analyzed.

Tea seed oil

Another oil that might be considered when identifying oils in Chinese lacquer is known as tea seed oil, pressed primarily from the large seeds of *Camellia sinensis* or *Camellia oleifera*. This oil is mentioned in the seventeenth-century Chinese literature on edible oil production (Song *et al.*, 1966). It is noted as a significant item of trade by European observers in the nineteenth century (Rondot, 1848; Hedde *et al.*, 1849) and continues to be so today (Ma *et al.*, 2011). It was mentioned in a late-eighteenth to early-nineteenth-century Chinese text as a cleaning agent for removing lacquer from filtering cloth and paintbrushes. We have found no further references in the Chinese literature to its use in lacquer production although it is specifically mentioned in the context of lacquer production by d’Incarville who wrote in Beijing in the mid-eighteenth century (de D’Incarville 1760) and again by Natalis Rondot in his description of lacquer production in Canton in the mid-nineteenth century (Rondot, 1848). D’Incarville stated that tea seed oil is a non-drying oil that is made siccative by boiling with arsenic. Recent analytical studies confirm that tea seed oil should be a very poor drying oil, with only 7–22% linoleic acid and negligible amounts of triple unsaturated fatty acids (Ma *et al.*, 2011; Wang *et al.*, 2011). The wide range of fatty acid compositions reported from different varieties and cultivars of *Camellia* suggests that the properties of tea seed oil might be quite variable. The Chinese lacquer makers that d’Incarville interviewed held that this was the only oil that would dry properly with lacquer. D’Incarville, sensibly, was skeptical of this claim. Samples of tea seed oil obtained by us through the open market did not cure or thicken appreciably within a month after being applied as thin films on glass. Attempts to thicken the oil and make it siccative by strongly heating and stirring with orpiment (arsenic sulfide) yielded no results after two days of continuous treatment. Further investigations with securely identified specimens of oil might shed some light on its use and possible processing.

Rapa oils

Another type of non-drying oil is also associated with lacquer making, specifically oils pressed from the seeds of *Brassica rapa* plants. This species contains many economically important subspecies including mustards, turnips and cabbages (many of the oil seed producing varieties were formerly designated as *B. campestris*). Seed oils from *B. rapa* were widely produced in China from antiquity. So-called white

cabbage oil (from *Brassica rapa* subsp. *pekinensis*) and rapeseed oil (primarily from *Brassica rapa* subsp. *oleifera*) are both listed as major Chinese agricultural products in the seventeenth century and are particularly recommended for cooking and lamp oil respectively (Needham et al., 1996). In the context of lacquer production, the saturated and non-drying rapeseed oil is usually mentioned in the literature as an oil for cleaning brushes and not for addition to lacquer (Webb, 2000). There are, however, occasional mentions of its use in direct association with lacquer manufacturing, such as its application for lacquer surface polishing in a late Yuan Dynasty text (Tao, 1959) and use as direct lacquer additive in Japanese literature (Sawaguchi, 1966, p. 144). In our laboratory, we prepared films of Japanese *roiro urushi* with an addition of 10% by weight of *rapa* oil. After curing at 80% RH for two days the lacquer had solidified but was exceptionally rubbery and flexible. Furthermore, the cured lacquer felt oily to the touch and had poor adhesion to the glass substrate.

Because it contains docosenoic acid, dried films containing *B. rapa* oil have unusually high amounts of tridecanedioic acid relative to the other dicarboxylic fatty acids (van Keulen, 2014). While rare in our analytical investigations, a small amount of *Brassica* oil seems to have been detected in the black lacquer layers of Chinese export panels from about 1700, now installed as architectural panels at Schoenbrunn Palace, Vienna, Austria. The identification was based on the enhanced relative amounts of high molecular weight dicarboxylic fatty acids and the presence of docosenoic acid (Schilling et al., 2016).

Obviously, more work could and should be done analyzing botanically secure specimens of the oils discussed above. Better statistical description of the expected ranges of P/S ratios might prove helpful, but further work to identify additional marker compounds that might help to differentiate these oils would also be very useful.

Proteins

Proteins are often added to ground layers of Asian lacquer as a binder. The three primary ingredients that are likely to contribute significant quantities of protein to Chinese lacquer structures seem to be pig's blood, animal glue, and egg. While Anacard lacquer is sometimes mixed with proteins in lacquer ground layers, this seems to be less common in Chinese export lacquerware. The addition of proteins to lacquer greatly increases the durability of ground layers and this may explain why the ground layers of Chinese export lacquers tend to fail (Piert-Borgers, 2000; Webb, 2000; Schellmann, 2012; Miklin-Kniefacz et al., 2014). A detailed discussion of the criteria for the identification of proteins in lacquer

objects using Py-GC-MS can be found in Schilling et al. (2016).

Pig's blood

Pig's blood is mentioned in the Chinese literature as an ingredient for use in Chinese lacquer foundation layers in records going back to the Yuan Dynasty (Tao, 1959). This material has a long tradition of use in mortars and plasters for architectural contexts (Zhao et al., 2015). In lacquer, pig's blood has been used as a primary binder or as a coating over the ground layer, especially in Chinese or Ryukyuan objects (Körber et al., 2016). In the course of our analytical investigations of a variety of Chinese lacquer objects, we have found blood to be a very common ingredient in Chinese ground layers, usually without the addition of Anacard lacquer. This accords well with descriptions given in a sixteenth-century Chinese text, preserved in Japan, known as *Kyushoku-roku* in Japanese (Arakawa, 1988) and *Xiushilu* in Chinese (Huang et al., 2004). The sixteenth-century commentator Yang Ming, whose annotations survive in the original text, stated that inferior substitutes that are easily breakable could be made with thick starch paste, pig's blood, 'lotus paste', and glue (without lacquer). In the early eighteenth century, Bonanni (2009, p. 23) also mentioned the use of pig's blood, mixed with quicklime, as a preparative coating for raw wood.

Animal glue

The relationship between lacquer and animal glue has long been close in Chinese culture. The *Spring and Autumn Period* (771–476 BCE) technology book lists lacquer and animal glue side by side in the list of materials required for manufacturing archery bows (Dai, 2003). In the early records there are no clear indications of the mixture of the two materials, but during the Northern Song Dynasty (CE 960–1127) we begin to find records of animal glue added to lacquer (Anon., 2001). In the course of our research using THM-Py-GC-MS, we have positively identified animal glue in ground layer samples from many lacquered objects; for details of the marker compounds used, see Schilling et al. (2016). To date, however, we have found no evidence of the mixture of animal glue into surface or decoration layers in the materials we have analyzed.

Egg

The addition of egg into lacquer shows up in a number of historic recipes that are compiled in a Ming Dynasty publication, which the main body of the text was dated to the Southern Song Dynasty (CE 1127–1279) (Tao, 1959; Yuan, 2006). In the majority of instances, the recipes call for the addition of egg white only, although in one recipe it is not clear whether whole

egg or white is specified. The recipes do not explain the purpose of adding egg to the lacquer, but a contemporary Japanese lacquer artist has suggested that it serves to thicken liquid lacquer (Kitagawa, 2010, personal communication). While we have identified several marker compounds for egg in lacquer based on published research and our own analysis of reference materials, egg has not yet been detected in our analytical studies of any Chinese lacquered objects.

Carbohydrates

Flour, of unspecified origin, is the primary carbohydrate-based additive cited in the Chinese lacquer literature, although it is rarely mentioned. Flour appears in a recipe dating to the Yuan Dynasty probably between 1329 and 1366 CE (Tao, 1959), as one of the materials used to in the making of a ‘ground layer’, but with no specific mention of the purpose of adding the flour. The addition of starchy materials to lacquer formulations is not uncommon in the Japanese lacquer tradition, where it is used to thicken the consistency of liquid lacquer (Kitano, 2005). In the course of our research, we have not yet detected starchy materials (other than the carbohydrates associated with laccol) in any Chinese lacquer, either in the surface or ground layers. Similarly, Schellmann, who studied and analyzed numerous lacquer foundation layers from objects in the Victoria and Albert Museum, did not report finding starch in any samples (Schellmann, 2012).

Wood distillates

Cedar oil

One of the most surprising and perplexing findings from our Py-GC-MS studies of Chinese lacquer formulations has been the remarkably frequent detection of cedrol and cedrene compounds. These compounds have been found in a wide range of Chinese lacquer objects, ranging from a Han Dynasty ear cup from Mawangdui, to an imperial Ming Dynasty plate bearing the Jiajing reign mark (CE 1521–1566), an eighteenth-century carved lacquer bowl from Yunnan and virtually all the pieces of late-seventeenth and eighteenth-century export lacquer that we have studied, including black, red, and coromandel lacquer. The presence of cedrol and cedrene suggest the intentional addition of some form of ‘cedar oil’ to the lacquer. Cedar oil is a poorly defined term which covers a wide range of oily and/or resinous materials extracted from the heartwood of trees in the family *Cupressaceae* (Langenheim, 2003, p. 329). Confusingly, virtually all, current and historical cedar oil production in China is from genera of *Cupressaceae* other than true cedars (*Cedrus* spp.); the best known source of cedar oil in China is the Chinese cypress (*Cupressus funebris*). Although the

taxonomy of the Chinese *Cupressus* is the subject of some debate (Adams & Li, 2008), it seems clear that there are several species and/or varieties of *Cupressus* that can produce chemically similar products. In addition, wood of *Juniperus* spp. (Juniper) and *Cunninghamia* spp. (Chinese fir) have also been shown to be capable of producing cedar oil (Adams & Li, 2008; Zhu, 2013).

Cedar oil is produced by two primary methods, either by steam distillation or pyrolytic distillation. Today, the term cedar oil normally refers to the steam-distilled product, which is a light-colored oily material of low viscosity that is used primarily in the perfume and fragrance industry. As mentioned above, we have detected cedrol and cedrene in Chinese lacquer ware from Mawangdui dating to the Han Dynasty. It is not entirely clear if the technology existed at this time to produce a refined product comparable to today’s cedar oil, although Needham *et al.* (1980, pp. 49–51) proposed that archaeologically recovered bronze artifacts from the early Han Dynasty (so-called rainbow vessels) could have been used for the sublimation and recovery of volatile substances, such as camphor, from wood. If true, such an apparatus might have been used to produce refined cedar oil.

The other common method of producing cedar oil is by pyrolysis, also called dry distillation, downward distillation, or destructive distillation. In this method the wood is stacked in a mound and then covered with earth or brick. The wood is burned slowly with very limited oxygen and the volatile and/or resinous components collect on the floor and run out of the mound via a sluice. As the process proceeds, the effluent changes from a thin, watery liquid to a progressively thicker and eventually tar-like substance. We have not yet found Chinese references to the production of cedar oil by this method, but in the west a very similar product called cade oil has long been produced from the wood of *Cupressaceae*, with the earliest Greek descriptions of its production dating to the third century BCE (Theophrastus, 1916, pp. 217–33). A detailed and highly relevant description of the production and uses of cade oil in modern North Africa is given by Julin who states that the material in common use is ‘a red-brownish to dark-brownish clear or turbid thick liquid’ (Julin, 2008). The primary uses of this material given by Julin are as a medicine or disinfectant, but it is also said to be used for the decoration of pottery.

A considerable amount of work has been done to determine the composition of both steam-distilled and pyrolytic cedar oil from a variety of species by chromatographic methods (Duquesnoy *et al.*, 2006; Adams & Li, 2008; Zhu, 2013). The compositions reported are quite variable and may depend on

factors such as the species or variety, the method of extraction, the temperature of extraction, and the fraction collected (early or late products of pyrolysis). Duquesnoy *et al.* state that fresh steam-distilled cedar oil is distinguished from the pyrolysis product primarily because the former contains large amounts of thujopsene in addition to α - and β -cedrene and cedrol, while the latter contains no thujopsene, but considerable amounts of α -, β -, and γ -eudesmol. Results published by Zhu (2013) would appear, however, to contradict this observation. In any case, none of the many samples of Chinese lacquer that we have analyzed appears to contain either thujopsene or eudesmol compounds. This state of affairs is difficult to interpret; the absence of thujopsene might indicate that pyrolytic oil was not used in Chinese lacquer, or it might simply be that any thujopsene present in our samples was decomposed by the pyrolysis that is part of our analytical method. A third possibility is that the thermal instability of thujopsene may mean that natural aging over hundreds of years can result in the near total loss of this compound. Clearly, further research on the subject would be valuable.

In our program of analysis we have consistently found that when cedar oil is present, cedrol appears to be more abundant than cedrene compounds. Most published analyses of cedar oil find the inverse to be true. The notable exceptions that we have found to date are isolated results from steam-distilled oils of *Cunninghamia* (Chinese fir) reported by Zhu (2013) and two species of *Juniperus* (Juniper) reported by Adams and Li (2008). This finding is suggestive, but it remains a possibility that cedrene compounds, which are more volatile than cedrol, are lost preferentially during aging. Additional work to understand the aging behavior of these compounds in lacquer could help to clarify the situation.

Notably absent in the Chinese or western literature we have surveyed is any mention of cedar oil being used in paints or varnishes, much less lacquer. Until further information is discovered, this leaves us able only to speculate about the form of cedar oil that might have been used and the reasons that cedar oil might have been added so frequently to Chinese lacquer formulations from ancient times until at least the eighteenth century. It is possible that a low-viscosity cedar oil was added to Chinese lacquers as a thinner or diluent to modify the working properties of the lacquer and increase gloss. Alternatively, a thicker and more viscous pyrolytic oil might have been used as an extender or adulterant. Either variety of cedar oil might also have acted as a plasticizer in the cured lacquer film. A final possibility that should be considered is that cedar oil was added as a preservative; raw lacquer is susceptible to spoilage due to bacteria or fungi and many traditional

medicinal uses for cedar oil depend on its antimicrobial properties, making it conceivable that it was added to increase the storage life of lacquer.

Camphor

Camphor exists both as a white waxy solid and as an oil, both commonly extracted from the wood of the camphor laurel (*Cinnamomum camphora*), although other related tree species can also be used. The use and production of camphor in China goes back to ancient times (Needham *et al.*, 1980); however, we have not yet found any mention of the use of camphor relating to lacquer in the Chinese literature that we have surveyed. There are, however, some mentions of its use in western sources and, as with tea seed oil, it was mentioned briefly in both de D'Incarville (1760, p. 137) and Rein (1889, pp. 149–50). Rein provided the details that 'a little' camphor was crushed before it is added to the lacquer, and that it dissolves in the lacquer, rendering it thinner. Another source in which we have found camphor mentioned is Quinn's famous nineteenth-century description of Japanese lacquer practice. In this work he described two types of lacquer prepared with the 'addition of one-third of camphor', or even more, and explained that it was added to thin the lacquer and make it easier to spread (Quin, 1882, p. 9). In more recent times, camphor has been used extensively as a plasticizer in nitrocellulose lacquers and it is likely that it would also have served this purpose in Asian lacquer films. In our analytical investigations we have detected camphor (based on the presence of camphor and camphene compounds) on only a handful of occasions. Camphor is, of course, a volatile material and thus it is possible that even if it was added, it might already have been lost from aged lacquer films.

Miscellaneous materials

Gall

Pig gall or bile is another material that is mentioned in several recipes for Chinese lacquer. Zhu and Gao (2002) cite a Qing Dynasty recipe for golden lacquer in which pig gall may be added as an alternative to camphor, with the purpose of thinning the lacquer and thus minimizing brushstrokes. D'Incarville also described the addition of pig gall (*fiel de porc*) at a concentration of three quarters of an ounce (5–6 gros) to one pound (*une livre*) of lacquer for the purpose of adding body to the lacquer (de D'Incarville, 1760, pp. 121–2). In the course of our research, bile (presumably of porcine origin) has been identified in a number of Chinese lacquered objects, including a late-eighteenth-century Chinese carved lacquer screen in the collections of the Weltmuseum Wien, Vienna, Austria.

Tea

We have thus far only found one textual reference to the addition of tea to lacquer and this comes from d'Incarville, who says that he once witnessed a Chinese lacquer worker add tea mixed with iron sulfate (*vitriol romain*) to lacquer, presumably to blacken it (de D'Incarville, 1760, p. 122). While we have yet to detect tea in any lacquer that we have analyzed, we regularly check for the presence of caffeine and have found this to be readily detectable in reference samples of lacquer that we have prepared with tea and iron sulfate.

Conclusions

In conclusion, this paper hopes to convey our growing understanding that Chinese lacquerware is compositionally much more complex and varied than previously understood. This may have significant implications for the further development of conservation treatments and may also offer new possibilities for dating, attributing, and authenticating Chinese lacquer. While our research to date has revealed certain interesting characteristics and components of Chinese lacquer, we consider that we have barely scratched the surface of a very complex subject, with a great many questions clearly remaining unanswered. Further studies and ever expanding collaborative research will doubtless expand our understanding of these unique and distinctive objects.

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